

EFFECTS OF RADIODARKENING ON THE LIGHT TRANSMISSION PROPERTIES OF YTTERBIUM-DOPED OPTICAL FIBERS

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Effects of Radiodarkening on the Light Transmission Properties of
Ytterbium-doped Optical Fibers

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March 2021

We certify that we have read this thesis and that in our opinion it is fully adequate,
in scope and in quality, as a thesis for the degree of Master of Science.

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ABSTRACT

EFFECTS OF RADIODARKENING ON THE LIGHT TRANSMISSION PROPERTIES OF YTTERBIUM-DOPED OPTICAL FIBERS

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M.S. in Materials Science and Nanotechnology

Advisor: Bülend Ortaç

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Optical fibers have been acting in a very crucial role in telecommunication and non-telecom industries like medicine, machining, sensing as well as in lasers and amplifiers when they are doped with rare earth ions for their remarkable features such as very good beam quality, high power generation and low cost after the fabrication of first low-loss optical fibers in early 1970s. However, these outstanding properties are deteriorated when optical fibers are exposed to ionizing radiation limiting their use in nuclear power plants and space applications. The main fabrication method of silica optical fibers, Modified Chemical Vapor Deposition (MCVD), as well as the effects of compositional variations of optical fibers on the gamma radiation resistance were highlighted by this thesis. Optically sound and robust fibers were evaluated through optical and chemical characterization methods and the light guiding abilities of them were measured before and after gamma radiation exposure to reveal its effects. Ytterbium-doped silica optical fibers showed significant transmission losses due to the creation of color centers by ionizing radiation exposure which were characterized and their contribution to radiation induced attenuation was described. Growth and recovery of these color centers at room temperature were analysed. Suggestions in terms of chemical composition for the fabrication of radiation resistant fibers as well as fiber dosimeters were made. The techniques that should be utilized for further recovery of the fibers were proposed.

Keywords: optical fibers, radiation induced attenuation, color centers, modified chemical vapor deposition, gamma radiation.

ÖZET

RADYOKARARMANIN İTERBİYUM KATKILI OPTİK FİBERLERİN IŞIK İLETME ÖZELLİKLERİNE ETKİSİ

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1970'lerin başlarında ilk düşük kayıplı optik fiberlerin imalatından sonra, çok iyi ışın kalitesi, yüksek güç üretimi ve düşük maliyet gibi dikkat çekici özelliklerinden dolayı optik fiberler, telekomünikasyon ve tıp, işleme, algılama gibi telekomünikasyon dışı endüstrilerde ve ayrıca nadir toprak elementleriyle katkılı oldukları zaman lazer ve amplifikatörlerde çok önemli bir rol oynamaktadır. Ancak, bu olağanüstü özellikler, optik fiberler iyonlaştırıcı radyasyona maruz kaldıklarında bozulur ve bu da nükleer santrallerde ve uzay uygulamalarında kullanımlarını sınırlar. Silika optik fiberlerin ana üretim yöntemi olan Modifiye Kimyasal Buhar Biriktirme (MCVD) tekniği ve optik fiberlerin bileşimsel varyasyonlarının gama radyasyon direnci üzerindeki etkileri bu tezde vurgulanmıştır. Optik olarak sağlam ve güçlü optik fiberler, optiksel ve kimyasal karakterizasyon yöntemleriyle değerlendirilmiş ve radyasyon etkilerini ortaya çıkarmak için gama radyasyonuna maruz kalmadan önce ve sonra ışık yönlendirme yetenekleri ölçülmüştür. Karakterize edilen ve radyasyona bağlı zayıflamaya katkıları açıklanan renk merkezlerinin oluşturulmasıyla iterbiyum katkılı silika optik fiberler radyasyona maruz kalmaya bağlı olarak önemli iletim kayıpları göstermiştir. Bu renk merkezlerinin oda sıcaklığında büyümesi ve geri kazanımı analiz edildi. Radyasyona dayanıklı fiberlerin yanı sıra fiber dozimetrelerin üretimi için kimyasal bileşim açısından önerilerde bulunulmuştur. Fiberlerin daha fazla geri kazanımı için kullanılması gereken teknikler önerildi.

Anahtar sözcükler: optik fiberler, radyasyon kaynaklı zayıflama, renk merkezleri, modifiye kimyasal buhar biriktirme, gama radyasyonu.

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Chapter 1

Introduction

Humans have used light as a tool for communication throughout history. John Tyndall exhibited that light can be transmitted through water in the 19th century, proving that the light can be bent using a media other than air. [1] This idea was developed by other scientists for years and resulted in the appearance of the first glass optical fibers in the 1950s, where a glass core surrounded with a layer that is thick enough was used to transmit light from one end to another. [1] The amount of optical losses then was quite a lot; thus, optical fibers were only feasible for short-range communication. However, in the 1960s, Charles K. Kao and George A. Hockham claimed that the optical fibers can be utilized as an efficient communication tool if the losses were below 20 dB/km, which was made possible by producing the fibers from high purity silica glass. [1] This idea brought Kao a Nobel prize in physics eventually in 2009. In the 1980s, the mass production of optical fibers with high efficiency became possible, which made optical fiber communication systems cheaper and feasible.

Optical fibers have gained global attention since they are pretty convenient in terms of high powers, exceptional beam quality, excellent optical damage endurance, phenomenal thermal management, low price, high optical efficiency, and low optical losses. [2–6] Thus, optical fibers found themselves in many application areas, including high-power lasers and amplifiers, communication, re- mote

sensing, material processing, military defense, and applied medicine. [3, 7, 8]

The performance of optical fibers in harsh environments on earth and in space attracted a lot of attention in recent years, which is mainly determined by the type of fiber, the elements doped in its core and clad, the radiation type that the fiber is exposed as well as the total dose amount and dose rate. It was shown that pure silica core fibers are much more resistant to gamma radiation than other fibers doped with elements like Germanium (Ge), Phosphorus (P), and Nitrogen (N). [9] This behavior is because the addition of other elements into the fiber leads to the formation of color centers under ionizing radiation, which is responsible for the creation of new absorption bands from ultraviolet (UV) to near-infrared (NIR) wavelengths. The optical loss caused by the absorption bands of color centers is called Radiation Induced Attenuation (RIA). The essential addition of metallic or non-metallic elements into optical fibers to reveal their optimum performance requires a deep understanding; therefore, much research has been conducted to exhibit their effect under gamma radiation. Many studies found out that the addition of Aluminum (Al), Ge, and P into Rare-Earth(RE)-doped, mainly Ytterbium (Yb) or Erbium (Er), optical fibers increases the optical losses caused by gamma radiation. [10–13] The best results related to the radiation resistance of RE-doped optical fibers were obtained when Cerium (Ce) was doped to these fibers. [14]

Another aspect of optical fiber and gamma radiation interaction is the use of fibers in radiation sensors. The first results exhibited that the optical fibers can be utilized in radiation sensing systems by adding the appropriate elements into their structure and making them sensitive to gamma radiation. [15, 16] These fibers have the advantages of working in broadband, transmitting signals with low optical losses, and being smaller in size and weight than the conventional radiation sensing tools.

Optical fibers can also be used for many different sensing applications, especially in medicine, thanks to their unique interactions with ionizing radiation. Some recent studies have shown that optical fibers yield quite promising results

when they are used for monitoring low-energy proton beams on a medical cyclotron [17], for temperature and pressure measurements in isotope production targets for nuclear medicine [18], for dosimetry in proton therapy [19], for neutron and proton beam sensor [20], for high-temperature sensing [21] and for atmospheric neutron monitoring [22].

In this thesis, the performance of three home-made Yb-doped optical fibers when exposed to gamma radiation was observed, and their comparison to a commercially available fiber was conducted. Radiation resistance of these home-made fibers, some of which are already used in kW-level fiber laser systems [23], were evaluated for the first time. This study's results would be very beneficial for the production of next-generations of fibers that can be used for high power signal delivery and radiation sensors as dosimeters in nuclear power plants as well as in space applications. The gamma-radiation exposure of optical fibers can also be conducted to provoke the formation of more defects, which are also formed during fabrication, to examine the effects of the defects that occur when the optical fibers are produced better.

Three home-made and one commercially available optical glass fiber, each having different compositions, were used in this study. All fibers were produced from silica glass cladding and silica core doped with Yb, Al, and P. Silica is a pretty ideal material for fiber production. It has many advantages in applications that require efficient light transmission due to its remarkable features like excellent (over 90%) light transmission from UV to NIR wavelengths, outstanding refractive index (RI) homogeneity, distinguished thermal stability and chemical durability, small thermal expansion coefficient, high mechanical strength and high resistance to radiation. Yb is doped to silica to take advantage of its optical activity to use these fibers in laser and amplifier applications.

Chapter 2

Theoretical Background

2.1 High-Power Fiber Lasers

Fiber lasers are highly demanded by scientific communities as well as medical and defense industries due to their astonishing features in terms of high average output power and high pulse energy. Figure 2.1 shows the improvements in both of these properties since the early 21st century are maybe the biggest reason why they are desired. [24]

Many benefits of the fiber lasers arise from dopants in fibers as well as fiber geometry. A very long length of fiber creates a huge path along which the light can interact with the active medium creating a quite large gain. In addition to the very good beam quality due to the single mode operation, fiber lasers can work at high average powers thanks to the very large surface active volume ratio of fibers which helps to dissipate the heat evolved during operation. Another good feature of fiber lasers is that the output beams of several fiber lasers can be combined to form one single beam which is the technique behind achieving very high average power fiber lasers as well as pumping closer to the emission wavelength and decreasing the quantum defect heating. By allowing to decrease the fiber length, this technique also contributes to the elimination of nonlinear

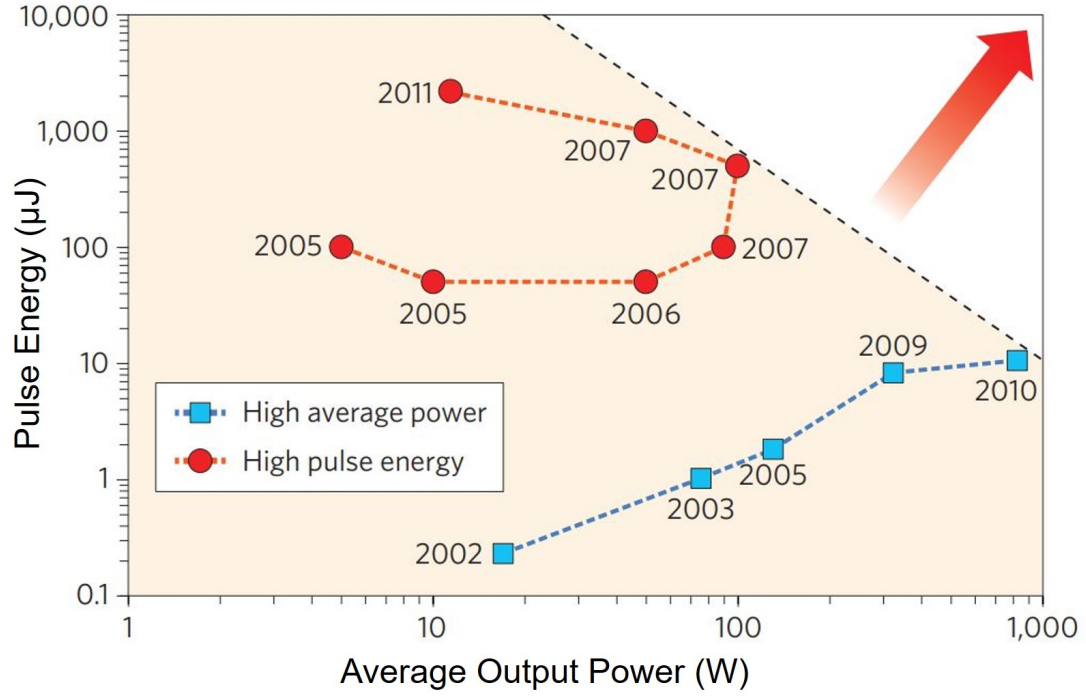


Figure 2.1: Enhancements in pulse energy versus average output power single-emitter femtosecond fiber lasers since 2002 [24]

effects that are the biggest drawbacks for the further improvements of fiber laser average powers.

Apart from their great benefits, fiber lasers have also some limitations some of which are still in question. These limitations occur when the high-power light is tightly confined within the very small dimensions of fibers and it propagates throughout their very long length creating the nonlinear effects which contains stimulated Brillouin scattering (SBS), stimulated Raman scattering (SRS) self-phase modulation, self-focusing and mode instability. Acoustic waves are also present inside the fibers and SBS stems from when the light propagating through the fiber interacts with them. Occuring above a power threshold, SBS decreases the average power and might damage the optical fiber or other optical elements. SBS can be mitigated by broadening the signal spectrum or by using fibers that does not allow the acoustic wave guiding. [25]

SRS occurs when the light interacts with glass lattice vibrations and it limits

the maximum output power. Techniques that can eliminate SRS include attenuating the scattered beam as well as using specialty fibers that do not allow propagation at the wavelength of the scattered beam. [26, 27]

The RI of the core of the fiber depends nonlinearly on the wavelength of the high intensity light that propagates through it. This nonlinear dependence is created by the Kerr effect that modifies the light wave phase by altering the path of the light. An instantaneous phase shift created by the Kerr effect changes across the pulse duration which is called self-phase modulation that reduces the optical efficiency. [28] Phase distortions which is the result of self-phase modulation can be eliminated by using a phase modulator.

Self-focusing is another result of the Kerr effect that leads to the creation of a nonlinear focusing lens in the fiber core which can damage the fiber by concentrating a high amount of energy to a small spot at high powers. Self-focusing can be prevented by changing the wavelength of the operation. [29, 30]

Mode instabilities occur above a certain average power threshold where normally stable high quality beams fluctuate. [31] It was found out that thermal effects result in periodic variations of fiber's refractive index profile (RIP) which create mode instabilities. [32, 33]

These harmful limitations caused by the nonlinear effects can be generally mitigated by reducing the light intensity that propagates through the fiber core. Increasing the core diameter; thus, the mode field diameter is an easy way for light intensity reduction. However, increasing the core diameter leads to other problems like guidance of higher order modes or an increase in bending sensitivity. Therefore, understanding fiber optics and fabricating appropriate optical fibers are key solutions to abolish the limitations of high power fiber lasers and to achieve further accomplishments.

2.2 Optical Fibers

Optical fibers are waveguides that can work at the wavelengths from UV to IR and they consist of a core and a cladding surrounding the core. The core has slightly higher RI than the cladding due to the effects of different various elements doped into core or cladding. This difference in the RIs enable optical fibers to be used as waveguides. [34] To increase their guiding properties as well as mechanical strength, the fibers are first coated with low refractive index polymer, called optical coating, and then with higher refractive index polymer, called mechanical coating.

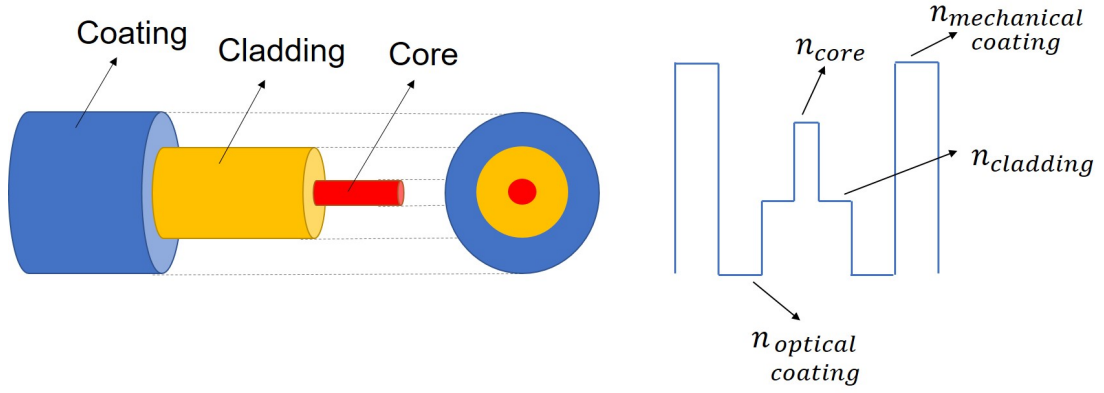


Figure 2.2: Optical fiber structure

The basic structure of an optical fiber can be seen above. The RIs of the core and cladding are denoted as n_{co} and n_{cl} , respectively. These indices depend on the wavelength of the light that propagates through them.

2.2.1 Acceptance Angle, Numerical Aperture (NA) and Relative Refractive Index Difference

Light propagates through the fiber by taking advantage of the Total Internal Reflection (TIR) principle. When the light is sent from the high index area to the low index area, the light fails to pass through the interface between and

is confined within the high index region. Snell's Law can clearly explain this phenomenon.

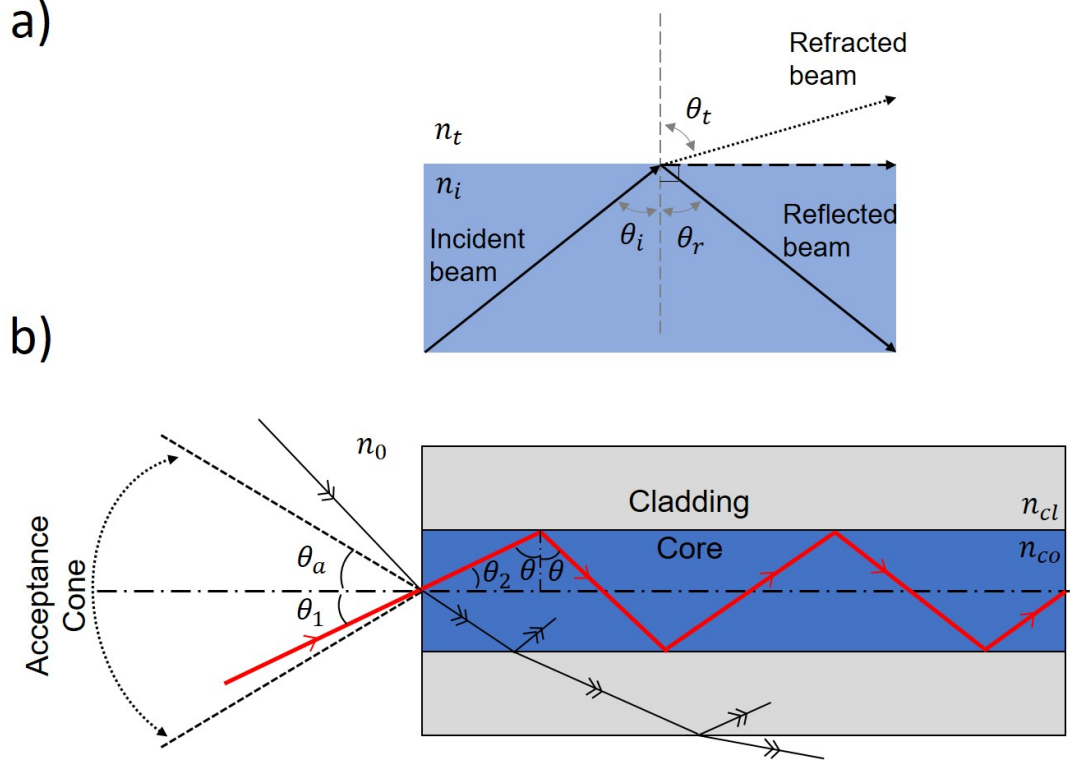


Figure 2.3: a) The paths of light passing from high index area to low index area ($n_i > n_t$) b) Propagation of light in an optical fiber

In Figure 2.3.a., it can be seen that when light passes from a high index area (n_i) to a low index area (n_t), its refraction angle (θ_t) is always bigger than its incidence angle (θ_i), obeying to the Snell's law. (2.1)

$$n_i \sin \theta_i = n_t \sin \theta_t \quad (2.1)$$

When θ_i reaches a critical value (θ_c), the light propagates alongside the boundary between the two regions and θ_t becomes 90° and $\sin \theta_t = 1$. Then, $\sin \theta_c = \frac{n_t}{n_i}$. At the incidence angles above this critical value, the light cannot pass through the low index area and reflects back to the high index area. This TIR principle establishes the foundations of light propagation in optical fibers with low loss. [35]

In order for the TIR to occur within the fiber, the light beam should enter the

fiber with an incidence angle that is less than acceptance angle (θ_a). In Figure 2.3.b., a beam with an incidence angle θ_1 enters the fiber at the interface of core and outside medium, say air (n_0). From the Snell's Law;

$$n_0 \sin \theta_1 = n_{co} \sin \theta_2 \quad (2.2)$$

Then, the reflectance angle of this beam from the core-cladding interface;

$$\theta = \frac{\pi}{2} - \theta_2 \quad (2.3)$$

where the angle θ is greater than θ_a . After that,

$$n_0 \sin \theta_1 = n_{co} \cos \theta \quad (2.4)$$

After the trigonometric substitutions,

$$n_0 \sin \theta_1 = (n_{co}^2 - n_{cl}^2)^{1/2} \quad (2.5)$$

If the medium outside the fiber is air ($n_0=1$), NA of the fiber becomes;

$$NA = \sin \theta_a = (n_{co}^2 - n_{cl}^2)^{1/2} \quad (2.6)$$

In other words, this equation expresses that any incident light satisfying $0 \leq \theta_1 \leq \theta_a$ conditions can be guided by the fiber. If $\theta_1 > \theta_a$ for the incident light, some of it will pass to the cladding and some will reflect. The NA parameter is defined as the fiber's ability to accept and propagate the light within the so-called Acceptance Cone, $2\theta_a$. Due to higher probability, light acceptance ability and transmission are high for the fibers having high NA. [36]

NA can also be stated by the RIs of the core, n_{co} and the cladding n_{cl} . The relative RI difference (Δ) defines the difference between n_{co} and n_{cl} . High values of Δ cause high scattering loss. [37] Thermodynamic fluctuations in dopant concentrations and irregularities at the core-clad interface result in this scattering loss.

$$\Delta = \frac{n_{co}^2 - n_{cl}^2}{2n_1^2} \approx \frac{n_{co} - n_{cl}}{n_{co}} \quad (2.7)$$

Then,

$$NA = n_{co}(2\Delta)^{1/2} \quad (2.8)$$

The fiber that guides more than one mode is called a multi-mode (MM) fiber whereas the one that guides only one mode is called a single mode (SM) fiber. Generally, $\Delta = 0.002$ for SM fibers and $\Delta = 0.02$ for MM fibers. [38]

2.2.2 Normalized Frequency (V)

Whether the fiber is MM or SM has tremendous importance in many different applications. Normalized frequency, or V number, defines the number of light modes that will propagate through the fiber. A small V number might lead fiber to be sensitive to cladding absorption losses, whereas a high V number may increase the scattering losses at the core-cladding interface. [39] Normalized frequency can be calculated as;

$$V = \frac{2a\pi}{\lambda}(n_{co}^2 - n_{cl}^2)^{1/2} = \frac{2a\pi}{\lambda}NA \quad (2.9)$$

where λ is wavelength of the light and a is radius of the fiber core. In order for a step-index fiber to operate in SM, $0 \leq V \leq 2.405$ condition should be satisfied. MM fibers can have higher V values.

2.3 Glass Optical Fibers

Glasses are amorphous, brittle materials that can conduct heat and be transparent to the light having a wavelength within UV, visible, or IR ranges depending on their composition. Although glasses can be fabricated from fluorides, phosphates, or chalcogenides, most commercial glasses are produced from silica (SiO_2). There are three types of SiO_2 glasses. One of them is quartz or silica made from natural materials and they have low ultraviolet transparency due to low OH^- concentration in their structure. Another one is fused silica produced synthetically by melting the silica and having low infrared transparency due to high impurity levels. The last one is clear fused silica which is a purified version of fused silica.

Figure 2.4 shows that the RI of SiO_2 increases with the amount of Al_2O_3 , P_2O_5 and GeO_2 doped, whereas B_2O_3 and F leads to a decrease. [40] Among many essential parameters that have to satisfy some optical fiber production standards, the RI of SiO_2 might be the most major one. It must have a RI value of 1.458 when guiding a light whose wavelength is 850 nm [41]; however, this RI value can be changed by adding other elements. The most commonly used

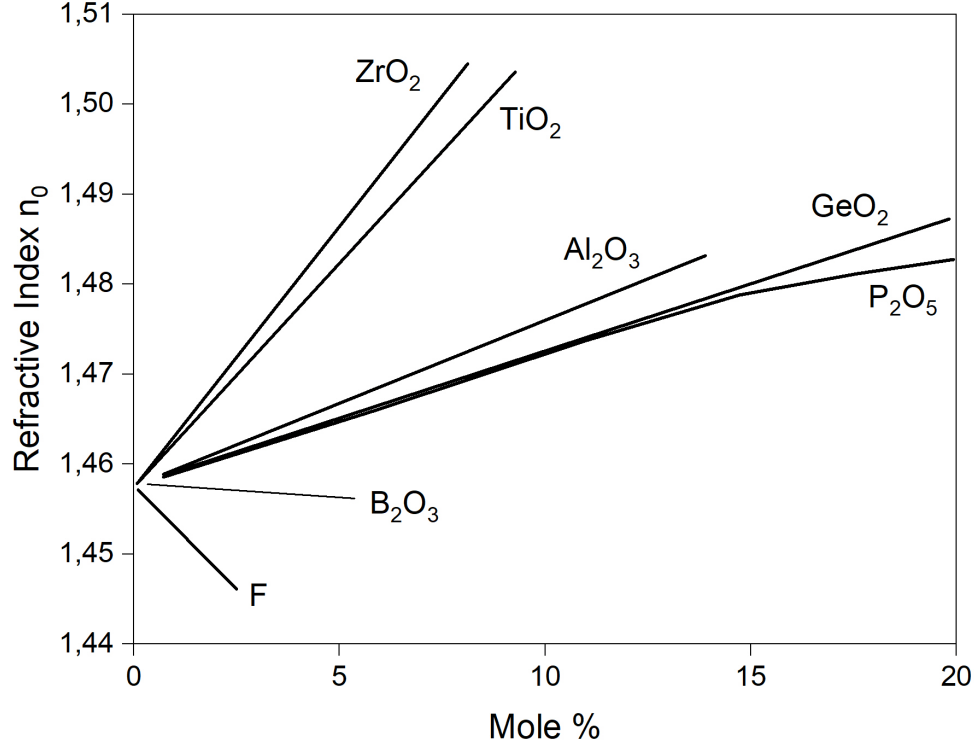


Figure 2.4: Effect of the additive amounts on the refractive index of silica [40]

oxides that are doped into fiber structure are Al_2O_3 , P_2O_5 , GeO_2 , F as well as some Rare-Earth (RE) oxides like Yb_2O_3 , Er_2O_3 , Tm_2O_3 for active fibers. [42] Therefore, the desired RIP suitable for required waveguiding properties can be obtained by doping the combination of these oxides into SiO_2 .

2.3.1 Step-Index Fibers

The RIP of step-index fibers is discontinuous and there is a noticeable difference between the RI of the core and the cladding where the former is greater than the latter as required for TIR. All the light modes having a smaller reflection angle than the critical angle are guided by the fiber.

The answer to the question of whether a step-index fiber is an SM or MM

fiber lies at the relative dimensions of the core and the wavelength of the light that propagates through the fiber.[17] If the core has similar dimensions to the light wavelength, the fiber supports the propagation of only one mode. If the core has larger dimensions, more than one mode is guided by the fiber. Thus, the SM fibers have much smaller cores than MM fibers. Also, intermodal dispersion does not occur in SM fibers since there is only one mode. Chromatic dispersion is the only dispersion in SM which is caused by material dispersion and waveguide dispersion. The former occurs because the RI of glass depends on the wavelength, whereas the latter is caused by the variation of group velocity with wavelength. The difference between the RIPs and mode propagation between SM and MM fibers can be seen in Figure 2.5.a and b.

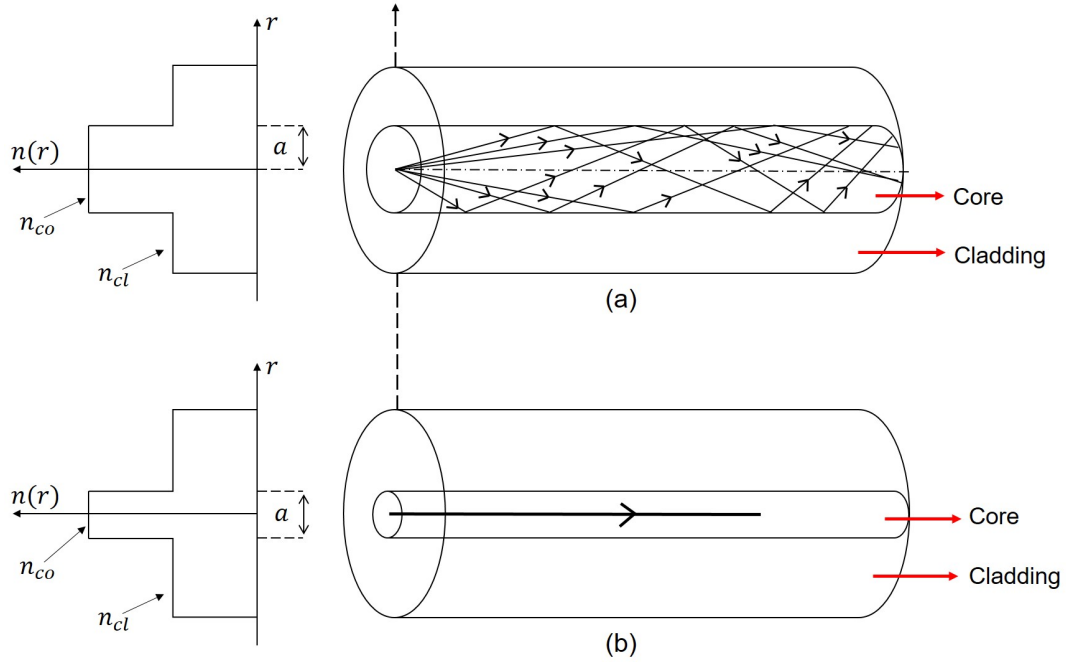


Figure 2.5: RIP and light propagation in a) Step-index MM fiber b) Step-index SM fiber

2.3.1.1 Ytterbium-Doped Optical Fibers

Active fibers are fabricated by doping Yb ions into silica glass as the gain material alongside Al and P forming the active fibers. When Yb-doped fibers are pumped

with the light at a wavelength of 915 nm, the pump light is absorbed by Yb ions. Then, the emission takes place at several wavelengths such as 940 nm, 980 nm, and above 1000 nm, depending on the stark splittings within the energy levels of Yb ions. The one at 940 nm has a lower intensity than the others; hence, it can be ignored. The main emission and gain for fiber lasers take place at 980 nm. [43]

The energy levels of Yb ions in silica glass can be seen in Figure 2.6.a. [43] Possible photon absorption and emission paths, as well as the photon wavelengths corresponding to these wavelengths, are shown. The lasing can be achieved when there are more ions at higher energy level $F_{5/2}$ than at lower energy level $F_{7/2}$ called population inversion. Absorption and emission spectra of Yb ions that stem from their energy levels are shown in Figure 2.6.b. [44] These spectra show the wavelength ranges of the light that can be absorbed and emitted. It also exhibits the maximum and minimum points within these ranges. Yb ions have different absorption and emission spectra when they are doped into a different matrix. To illustrate, the emission and the absorption spectra of Yb ions in Yb-doped yttrium aluminum garnet (YAG) crystals ($\text{Yb}^{3+}:\text{YAG}$) are entirely different from the ones shown for silica matrix in Figure 2.6.b. [45]

Optical fiber types include the ones mentioned above, but they are not limited by them. Various other fiber types can be produced, each having different features. Some of the other fiber types are graded-index fibers and specialty fibers whose cross-sections are microstructured like photonic crystal fibers (PCF) or polarization maintaining (PM) fibers.

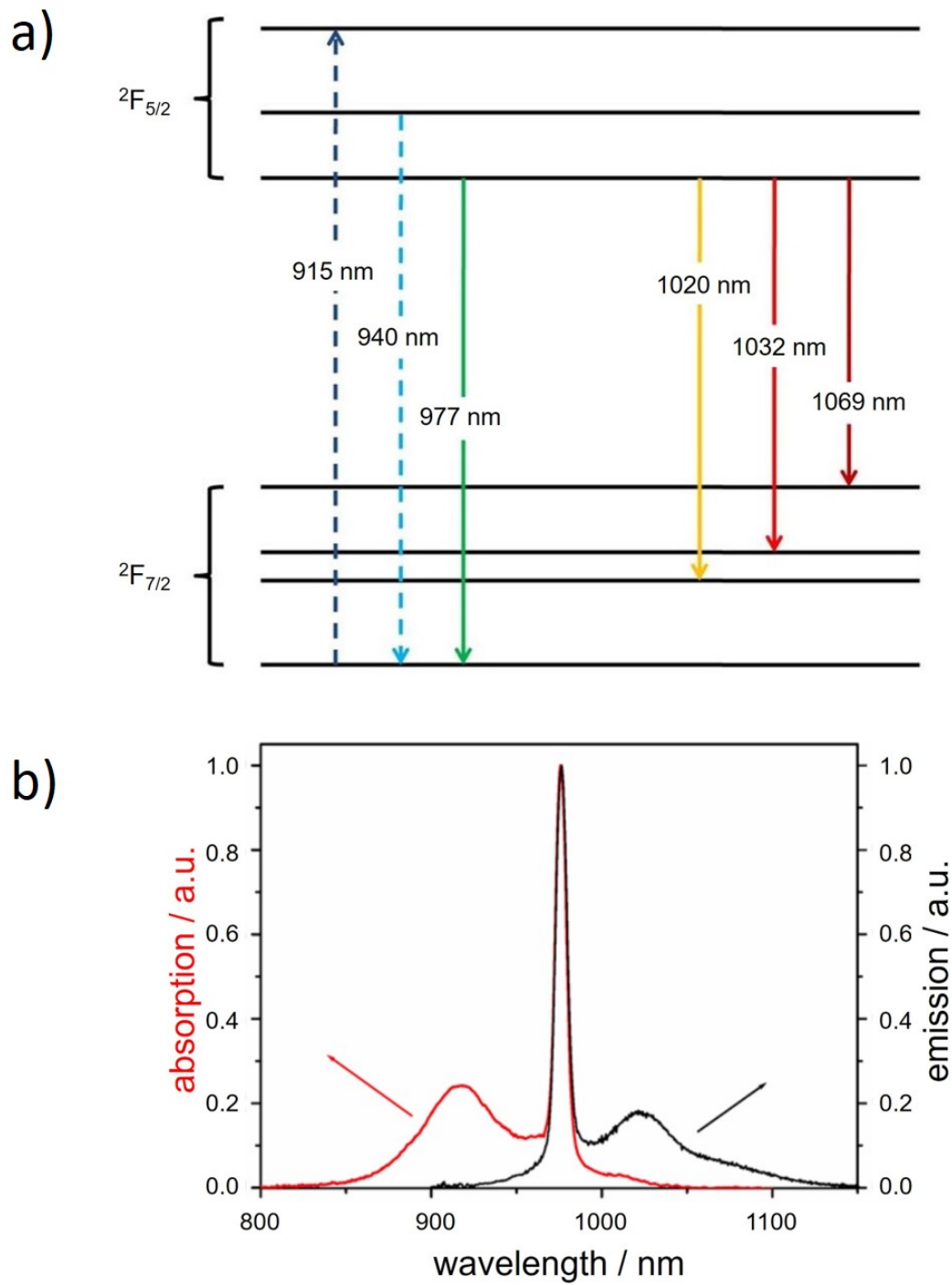


Figure 2.6: a) The energy-level diagram of the Yb ions in silica [43] and b) Absorption and emission spectrum of Yb in silica [44]

2.4 Fiber Losses

Absorption, bending, and scattering losses are the primary transmission loss mechanisms occurring in optical fibers. They are significant features of fiber optics due to their effects on output optical power, data transmission speed, and system efficiency. The measure of the loss is called attenuation. The changes in the power of the light while its transmission through an optical fiber is governed by the Beer-Lambert law [46];

$$\frac{dP}{dz} = -\alpha P \quad (2.10)$$

where α is the attenuation coefficient. Then,

$$P_{output} = P_{input} \exp(-\alpha L) \quad (2.11)$$

where L , P_{output} and P_{input} are the fiber length, power of the light at the end of the fiber and power of the light when it is coupled into the fiber, respectively. Thus, the attenuation is calculated as;

$$\alpha(\lambda) = \frac{10}{L} \log \frac{P_{input}}{P_{output}} \quad (2.12)$$

2.4.1 Absorption Losses

The fiber itself absorbs a portion of the light propagating through a fiber due to several reasons. One reason is the formation of atomic defects in the fiber structure during fiber fabrication. After the optical preform is produced, it is drawn into the fiber at the drawing tower by placing the preform into a furnace and heating it above the glass's softening temperature, during which the atomic bonds within the fiber are ruptured and then solidifies rapidly at room temperature. Thus, atoms form imperfect bonds in these nonequilibrium conditions. Furthermore, the clustering of Yb ions and misplacement of elements like Al or P during the above process contributes to absorption losses. Another reason is that the addition of metallic or non-metallic elements into fiber to optimize its performance leads to additional absorption bands. Additive elements like Yb, Al,

P, or the OH^- ion increase the amount of light absorbed at specific wavelengths. Finally, the material itself that the fiber is made from causes absorption loss. Either made from glasses like silica, fluoride, chalcogenide, or a polymer, fibers are designed to work with high efficiency and low losses at specific wavelengths and high loss for the rest of the spectrum. All of these base materials have their absorption bands somewhere in the optical spectrum and; hence, they lead to absorption losses.

2.4.2 Bending Losses

Optical fibers are not always used straight. Sometimes they need to be bent or coiled during operation. When the fiber is bent more than its limits, the conditions for TIR are not satisfied. Thus, some portion of the light escapes from the core into the cladding, which eventually leaves the fiber completely. Therefore, overly bent fibers experience increased loss. This loss mechanism is described in Figure 2.7. where a fiber partially loses its waveguiding mechanism due to extreme bending and some of the light rays escape outside of the fiber. In some cases, higher-order modes are more sensitive to bending than the fundamental mode. This situation can be taken advantage of by eliminating the higher-order modes and obtaining a single-mode operation which is the fundamental mode.

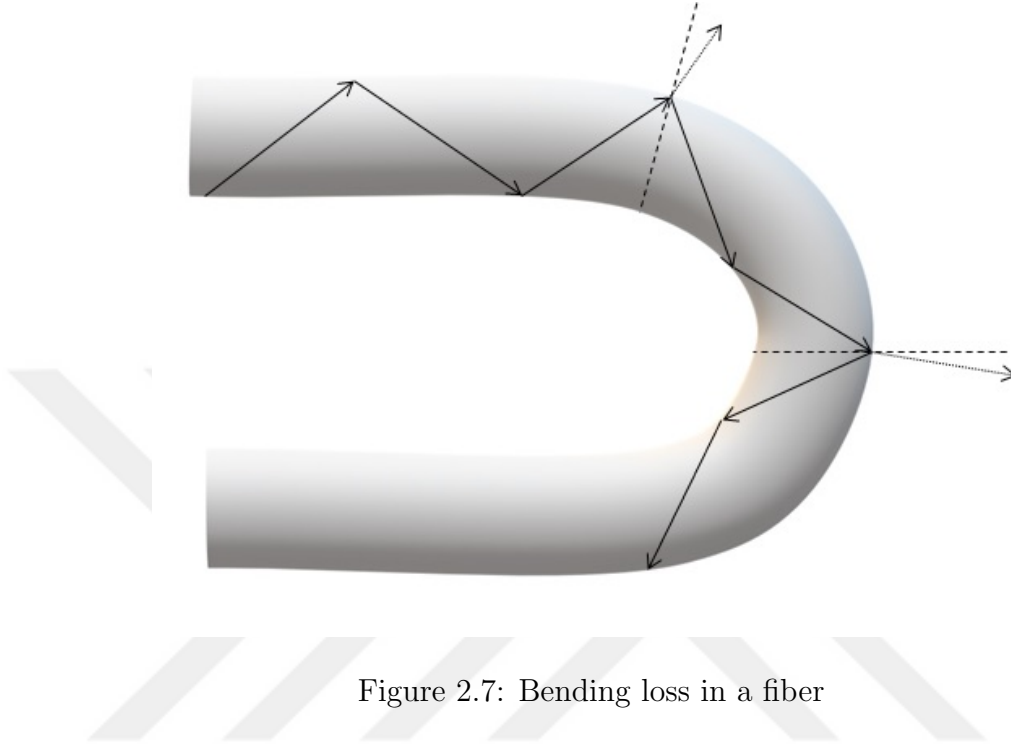


Figure 2.7: Bending loss in a fiber

2.4.3 Scattering Losses

During fiber fabrication, minimal variations in glass density, composition, and RI, additive element clustering, misplacement of the atoms might occur. The light may scatter from these inhomogeneities, causing additional loss. Rayleigh scattering occurs when the light scatters due to an inhomogeneity with a size smaller than the wavelength of the light. This loss is proportional to $\frac{1}{\lambda^4}$ and it decreases as the wavelength increases. [35] Rayleigh scattering is dominant for the light having a wavelength smaller than 1000 nm.

Figure 2.8 reveals the dominant loss mechanism in a SiO_2 optical fiber over a broad spectrum. Losses at the UV-end of the spectrum are caused due to the stimulation of electrons to a higher energy level by absorbing the photon energy. However, the losses at the IR-end are caused by the absorption of the light by the Si-O bond corresponding to their vibration frequencies in silica fibers. The presence of OH^- ions creates an absorption band at around 1380 nm causing a significant loss.

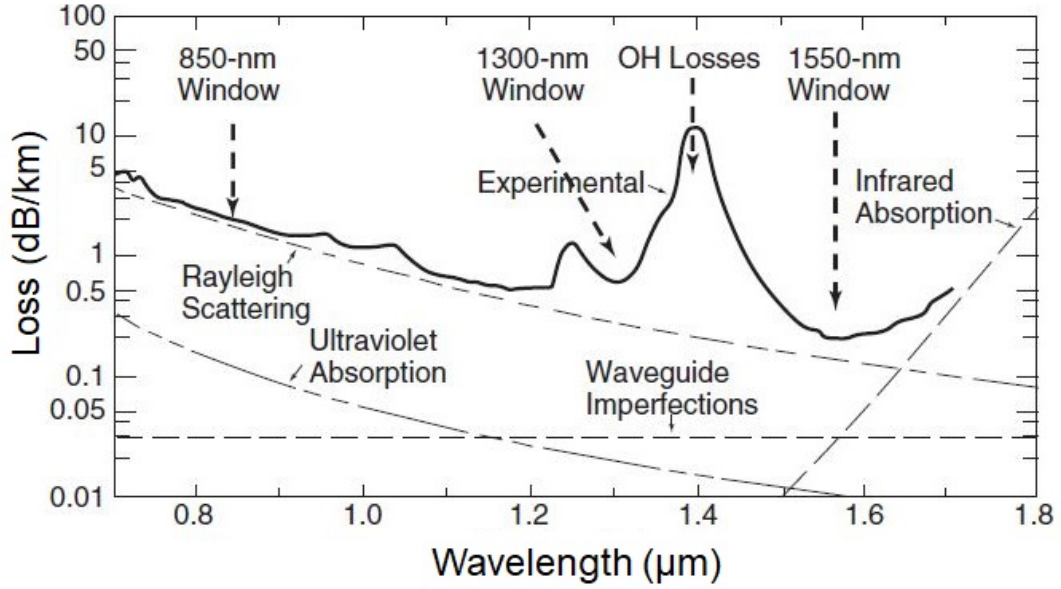


Figure 2.8: Loss spectrum of a SiO_2 fiber [34]

2.5 Optical Fiber Fabrication

Optical fibers have been produced all around the world by many various companies since the early 1970s. Their fabrication process is composed of two main steps: the production of the optical preform and fiber drawing.

2.5.1 Optical Preform Production

Optical preforms can be regarded as the macro-size version of an optical fiber and their manufacture is the first step within the optical fiber fabrication process. Although there are various methods such as Outside Vapor Deposition (OVD) or Vapor-Phase Axial Deposition (VAD), Modified Chemical Vapor Deposition (MCVD) is the most commonly used technique to produce an optical preform since its first description by MacChesney *et al.* in 1974. [47] This process enabled the realization of easy, high purity, low-loss optical fiber production. To mass-produce the optical fibers that have the best optical, mechanical, and geometrical

properties, fabricating optical preforms by MCVD as well as MCVD itself have been studied by many scientists for over four decades.

The fabrication of silica optical preforms in MCVD includes the deposition of high purity materials on the inner wall of a silica substrate tube and then collapsing this tube to an optical preform rod ready for subsequent fiber drawing.

The first step involves the evaluation of the substrate tube. This tube has to be produced as pure SiO_2 without any impurities. In addition, its dimensions and composition should not vary over its length and radius for high-quality preform fabrication. Then, the tube is placed into a rotating glassworking lathe. The substrate tube entrance is connected to a chemical delivery system from where the raw materials are sent in for the deposition, and the exit is attached to a larger tube that collects the unreacted and undeposited chemicals as well as by-products and takes them to the chemical scrubber system. The MCVD process scheme can be seen in Figure 2.9.

The most commonly used precursors in MCVD include halides like SiCl_4 , GeCl_4 , POCl_3 , AlCl_3 , SF_6 and SiF_4 . The reason why halides are used is that they have much higher vapor pressures than the metal impurities that might be present in the dopant sources. These impurities are left behind when the carrier gases transport the precursors into the substrate tube. Therefore, incorporating these impurities into fibers, hence, optical losses, is prevented by this technique, which acts as a purification process. The choice of these precursors and their usage extent depend on the type of elements desired to be doped in the core and the cladding of fibers. For active fibers, solid chelates of RE metals such as $\text{Yb}(\text{thd})_3$ or $\text{Er}(\text{thd})_3$ are also used to be doped in the fiber core. The precursors are sent into substrate tube in gaseous form along with carrier gases like O_2 and He by the chemical delivery system. SiCl_4 and POCl_3 are kept in the liquid phase in the bubbler at around 35°C and they are sent into substrate tube in gaseous form by O_2 and He carrier gases. AlCl_3 and Yb chelate are placed in the MCVD reactor in the solid phase and they sublime at 135°C and 185°C , respectively. Then, they are sent into the substrate tube, where the vapor phase reactions take place at around 1600°C and the precursors oxidize to form solid particles.

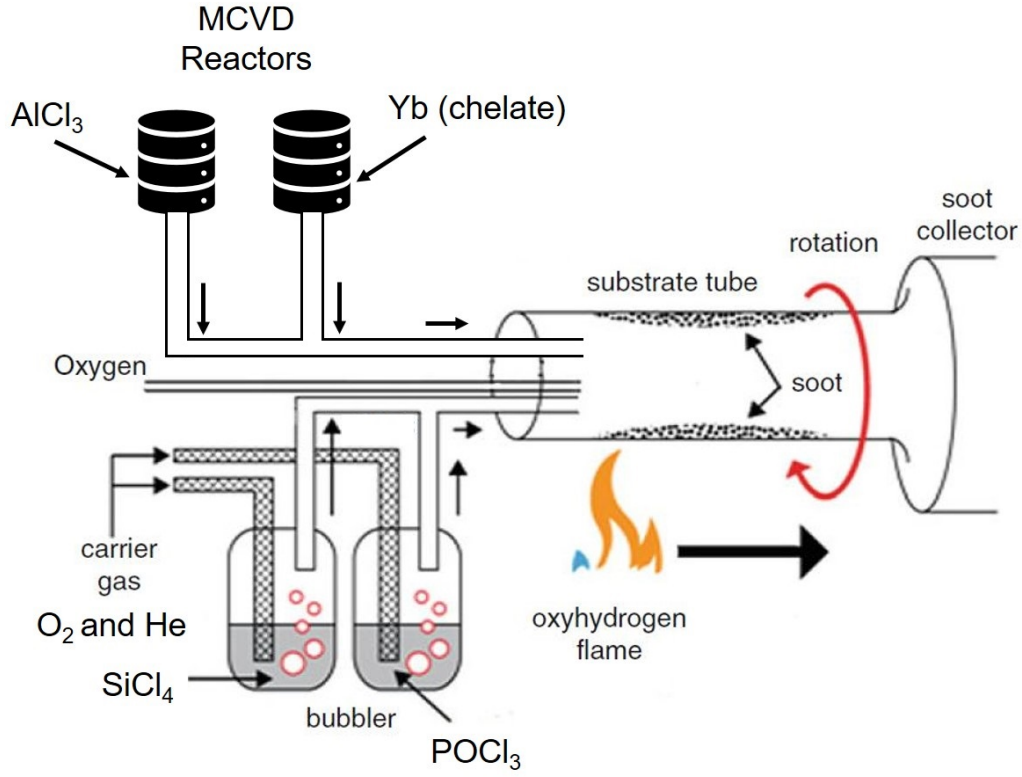
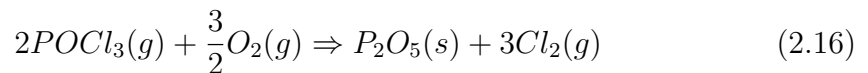
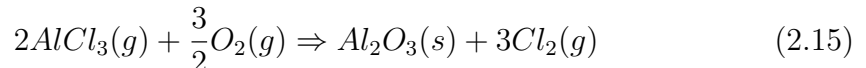
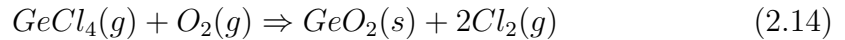
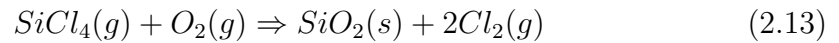
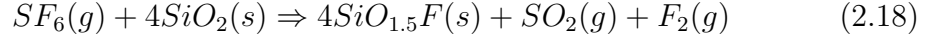
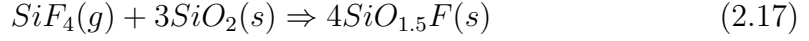


Figure 2.9: Modified Chemical Vapor Deposition process scheme [Adapted from [48]]

The substrate tube is heated with an oxyhydrogen torch traversing along the tube length. After the precursors are sent into the tube, they react with O_2 at elevated temperatures to form the solid oxide particles of each element which eventually deposit on the inner wall surface of the substrate tube and form soot. After the soot formation, subsequent heating of the tube by the traversing oxyhydrogen torch results in the sintering of the soot and formation of the glassy material. Some of these vapor phase reactions and the formation of their solid products are given below:





The desired fiber composition and final RIP determine the dopant types and precursor amount that is sent to the system. Although the cladding is mostly pure silica, dopants like F or P_2O_5 can be doped as well. In comparison, the fiber core consists of silica doped with Al_2O_3 , P_2O_5 , GeO_2 as well as with Yb_2O_3 or Er_2O_3 for active fibers. The cladding and the core are formed layer-by-layer deposition of these dopants and the final RIPs are created. The effects of these dopants on the RI of silica are shown in Figure 2.4. The most common types of RIPs can be seen in Figure 2.10. below.

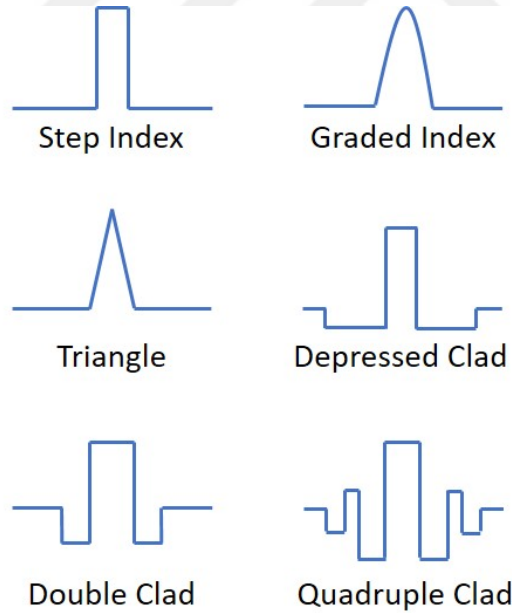


Figure 2.10: Common fiber RIPs

There lies a temperature gradient within the tube when heated by the oxyhydrogen torch, which results in the formation of a reaction zone where the temperature is ideal for the gaseous phase reactions (Figure 2.11.a.). The oxidation of $SiCl_4$ and $POCl_3$ are completed above $1400^\circ C$. After the formation of the solid particles, a phenomenon called thermophoresis is responsible for their deposition on the substrate tube's inner surface. These particles experience a net driving force that moves them towards the lower temperature region since the

gas molecules hitting the particles from the hotter side have more kinetic energy than those from the colder side (Figure 2.11.b.). Therefore, the solid particles end up incorporated onto the inner surface of the tube.

When the solid particles are first formed, they move inwards because the gas stream is colder than the substrate tube. However, the tube is colder than the gas stream away from the torch, making the particles move towards the tube and deposit, eventually. Although many of the reactions listed above have 100% efficiency, that is not the case for the deposition. The deposition efficiency E is described by

$$E \approx 0.8 \left(1 - \frac{T_e}{T_{rxn}} \right) \quad (2.19)$$

where T_e is the temperature at which the gas stream and the tube wall equilibrate and T_{rxn} is the temperature at which the chemical reactions that form the solid particles occur. T_e depends on the parameters like torch traverse length and speed, the thickness of the substrate tube wall, tube radius, ambient temperature, and the gas stream's flow rate. [48, 49]

The deposition of the particles occurs over a certain length and this length depends on the total volumetric flow and the thermal diffusivity of the gas stream. [49] As shown in Figure 2.11.b., not all the particles deposit. The ones formed near the tube wall have a higher chance to deposit than the ones forming near the center of the stream. The particles formed at the center deposit further away from the torch, if they do at all. The undeposited particles follow the streamflow and go to exhaust.

The solid particles formed in MCVD are several angstroms in size initially, but the larger aggregates are created by the collision of these particles to each other. These aggregates eventually sinter together. The final size of the particles depends on the glass composition and temperature. The coexistence of certain dopants might affect each other's deposition dynamics, which is the case for P and Ge. The presence of POCl_3 affects the deposition position and the composition of GeO_2 particles. [50]

After the deposition of dopants to form fiber cladding and core, the collapse

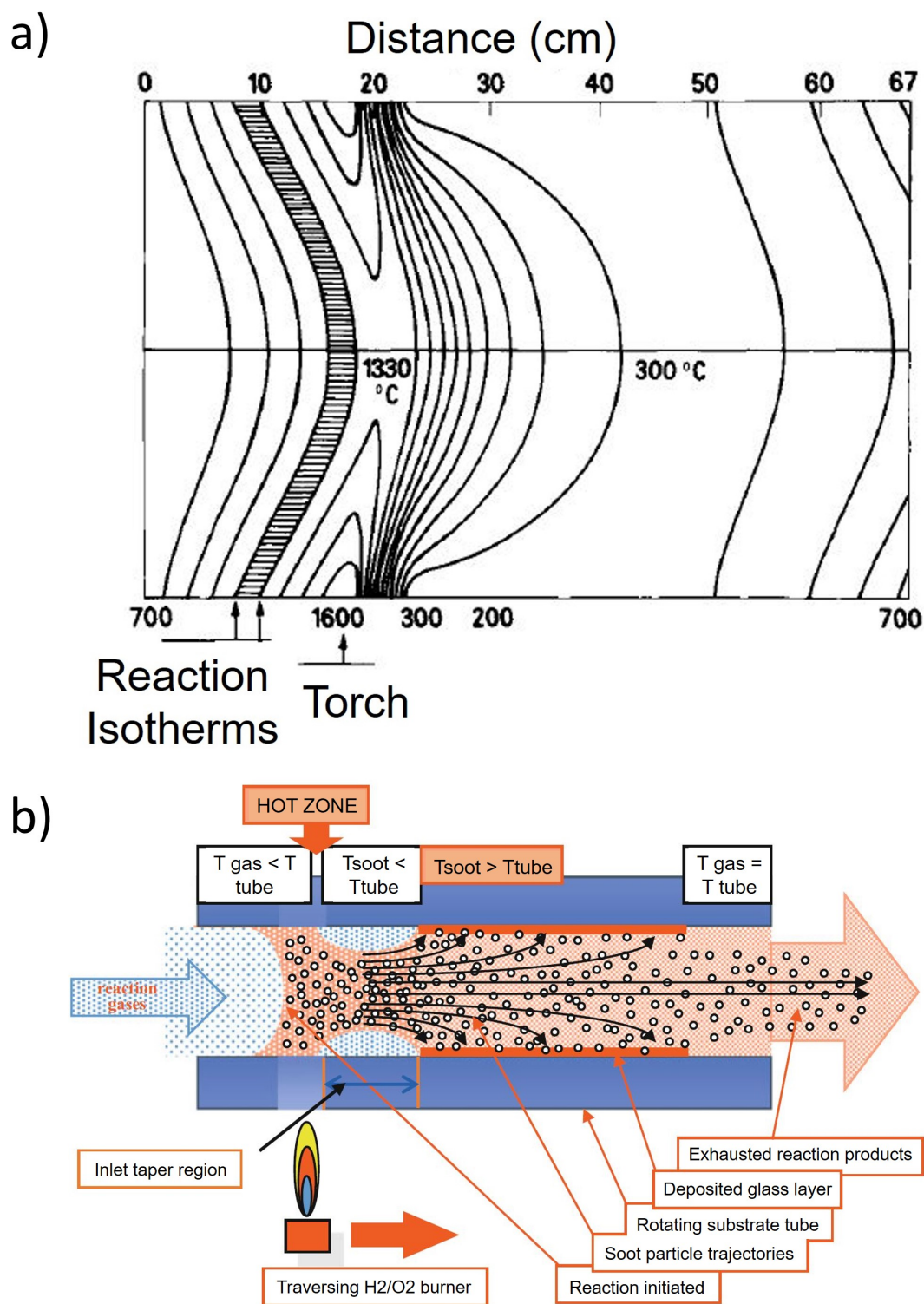
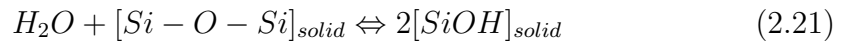
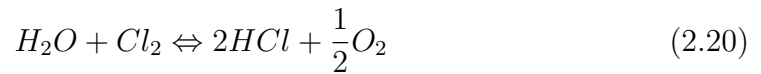


Figure 2.11: a) Temperature field within the MCVD substrate tube [50] b) The deposition mechanism and particle trajectories resulting from temperature field [48]

stage begins, which includes transforming the substrate tube into a preform rod by taking advantage of the high temperatures and the pressure difference between inside and outside of the tube. The tube's temperature is increased to around 2000°C by the oxyhydrogen torch or a furnace and the gas flow inside the tube is decreased so that the pressure outside of the tube will be greater than that of the inside and the tube will collapse inwards. The collapse behaviors of SM and MM fibers are different since the different dopants result in different viscosities. The viscosity and thickness of the total deposition affect the collapse rate significantly. The high temperature at the collapse stage might cause the evaporation of the P_2O_5 that is doped into fiber and may alter the RIP. One technique to prevent the evaporation of the P_2O_5 from happening is to feed the system with $POCl_3$ during the collapse so that the additional P_2O_5 deposition will compensate for its evaporation. [51] Another technique might be that the exit end of the substrate tube is collapsed first so that the evaporated material stays inside the tube during the rest of the collapsing process.

All the impurities should be got rid of to produce high-quality, low-loss optical fiber. Transition metal impurities that may present in the precursors can be avoided using halide precursors, as mentioned earlier. However, the amount of water in fibers should be minimized since it causes additional losses between 1.3-1.6 μm region where the intrinsic losses of silica are minimal (Figure 2.8). The main Si-OH absorption peak is at 2.7 μm , but it has overtones in the NIR region. In addition, the P-OH absorption band peaks at 1.6 μm and it increases with SiOH and P_2O_5 content. [52]

The two main OH sources are the hydrogen impurities that reside in the precursors or enter the gas stream from a leak and the hydrogen that is present in the starting substrate tube. Water presents in glass as Si-OH and the OH incorporation into fiber is controlled by chlorination. [53, 54]



Then, it was shown that the OH content in glass can be expressed as:

$$C_{SiOH} \propto \frac{[P_{H_2O}^i][P_{O_2}]^{1/4}}{[P_{Cl_2}]^{1/2}} \quad (2.22)$$

where $P_{H_2O}^i$ is the initial partial pressure of H_2O in the gas stream coming from all sources like HCl or $SiHCl_3$ and P_{O_2} and P_{Cl_2} are the partial pressures of O_2 and Cl_2 . [55]

Figure 2.12. shows the relation between the OH incorporated in glass and oxygen and chlorine partial pressures when there is 10 ppm H_2O equivalent in the gas stream in MCVD. [55] It was found out that there are 3-10% Cl_2 present in the system during deposition due to the oxidation of halide precursors and most of the hydrogen is converted to HCl , which does not end up in the glass. Therefore, the amount of OH incorporated into the glass is lowered by a factor of 4000. A significant amount of OH might end up in glass if no Cl_2 is present in the system. Thus, feeding the system with Cl_2 can help immensely to eliminate the hydrogen impurities in the glass. To decrease OH inclusion in preforms, these equilibrium conditions offer three suggestions: preventing the hydrogen contamination, including the starting materials and system leaks, lowering the partial pressure of oxygen, and raising the partial pressure of chlorine.

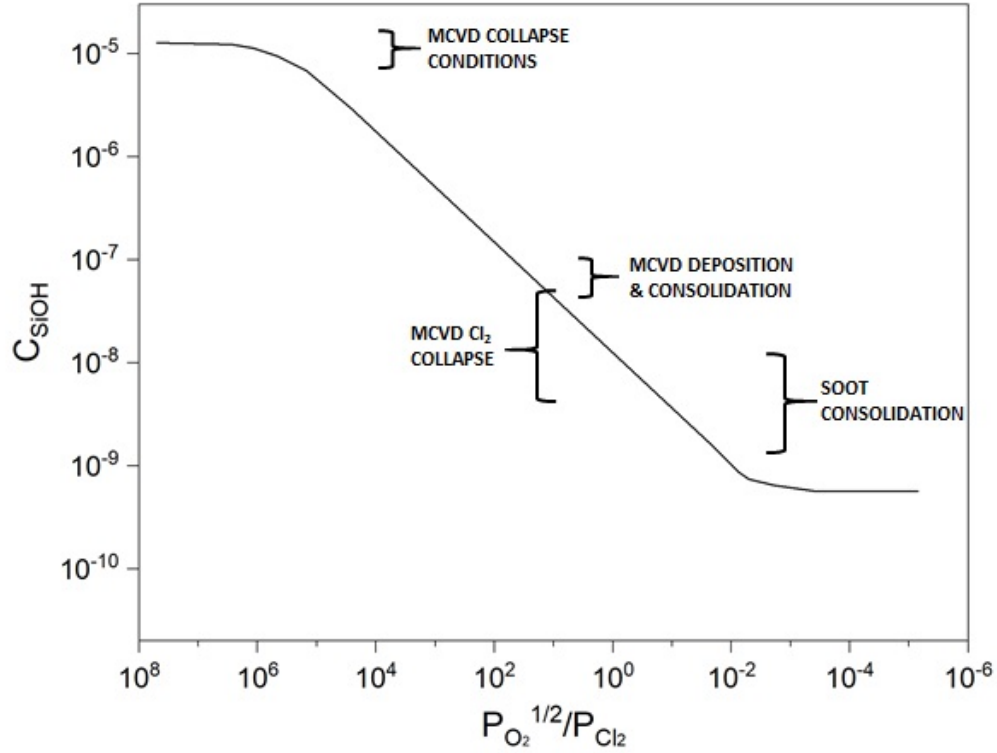


Figure 2.12: SiOH incorporation relation with oxygen and chlorine partial pressures with 10 ppm H_2O in the starting gas [50]

Some suggestions were made to keep the hydrogen incorporation at the minimum. The precursors, the system, and the substrate tube should be made free of the ambient atmosphere's humidity. Furthermore, all the connections of the MCVD system should be leak-free. The precursors must be as pure as possible to fabricate a low-loss fiber.

Decreasing the partial pressure of oxygen can be obtained by adding inert gasses such as He or Ar to the O_2 carrier gas. It was mentioned that there are %3-10 Cl_2 in the MCVD deposition stage and it was argued that increasing this value alters OH inclusion greatly. In addition, to prevent the OH from coming from the substrate tube, the tubes with a low amount of OH should be purchased or produced if possible. If the substrate tube contains high OH, thicker claddings should be deposited to eliminate the OH-related losses. [50]

Elimination of bubbles that might form within the optical preform is another critical concern since they contribute to the total loss. Parameters like temperature, glass composition, and viscosity, the sintering rate should be considered thoroughly. Forming and depositing large aggregates as well as dopant vaporization should be avoided to eliminate the bubble formation.

After the preform is produced, it is evaluated by many various characterization techniques to check its physical condition, dopant concentration, RIP, impurities, and bubbles. If all the limitations are satisfied, the preform is ready to be drawn into fiber.

2.5.2 Fiber Drawing

After the desired optical preform is fabricated, its cylindrical shape is changed into hexagonal or octagonal shape using a computerized numerical control (CNC) lathe. Changing the fiber shape from cylindrical to the octagonal improves cladding light absorption efficiency from 10% to around 90% that enhances the energy transfer into RE ions in the core since all the light rays in the cladding eventually crosses the core, which is not the case for the cylindrical fibers. [56] Then, the preform is drawn into fiber in a fiber drawing tower whose basic scheme can be seen in Figure 2.13.a. In this process, the fiber is placed into a preform feeder platform that shoves the preform into a graphite resistance furnace where the preform is heated above its softening temperature around 2000°C. With the help of the gravitational force, the softened preform starts to flow downwards in the form of a big raindrop and it gets thinner to turn into a fiber. Eventually, it is caught by the capstan, which pulls the fiber in order for it to be coiled in the winding machine. The fiber's diameter depends on the preform feeding speed and fiber pulling speed by the capstan. If the fiber diameter deviates from the desired value, fiber pulling speed can be changed from the control unit. The drawing temperature and the drawing tension are important parameters that influence the final fiber shape and the mechanical stress that resides in the fiber. It was found out that the octagonal shape of the preform is lost during drawing and the

fiber becomes circular if the drawing temperature is too elevated. In addition, the fiber can become fragile if the drawing tension is too high. Therefore, the optimum values of the drawing temperature and tension, as well as the fiber pulling speed, should be determined for the desired features of the final product.

The first polymer coating has a lower RI than the fiber cladding and it is called the optical coating. It enables the light to be guided in the cladding by forming an index difference between itself and the cladding so that TIR can occur similar to that between the core and the cladding (Figure 2.3.b.). When the fiber is coated with a low RI UV-curable polymer, it is named double-cladding fiber since the optical coating acts as the second cladding. In double-clad fibers, the light can be guided through the cladding and the light eventually crosses the core, where the optically active RE ions absorb it. The lower the RI of the optical coating, the more energy transfer to RE ions from the light pumped into the cladding.

The second polymer coating is called the mechanical coating, which protects the fiber from mechanical damages and increases the fiber strength as well as its bending ability.

The cross-section image of the finalized fiber under an optical microscope can be seen in Figure 2.13.b. which reveals the final shape of the fiber product as well as the parts it consists of. The brighter small region at the center is the fiber core which is surrounded by the cladding. The dark coating is the low RI UV-curable polymer coating and the outer yellow one is the mechanical polymer coating. The diameters of the core and the cladding were 26 and 410 μm , respectively. [23]

After the fiber is fabricated, it is tested through optical, elemental, and mechanical characterization steps. The fiber is ready to be utilized if all the test criteria are met.

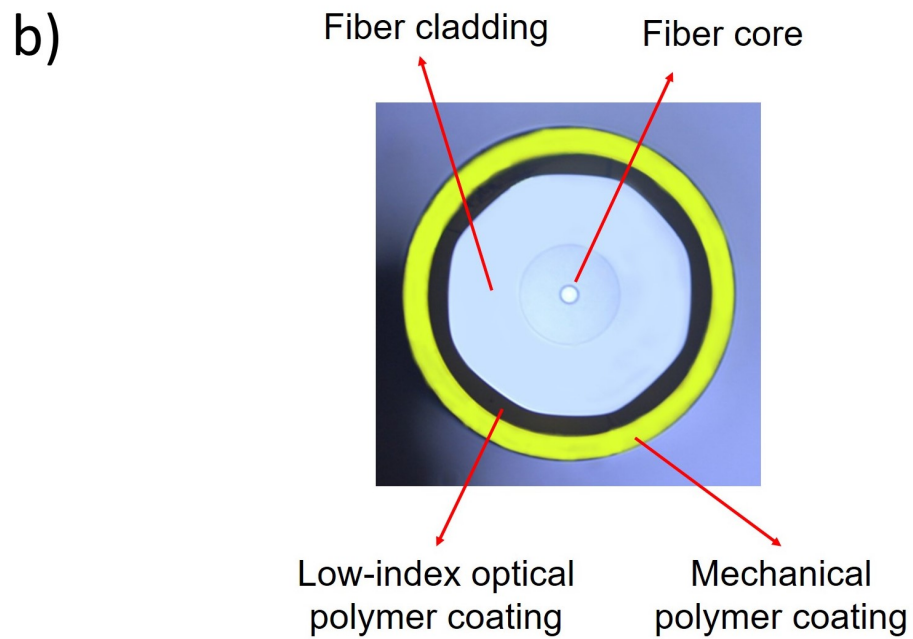
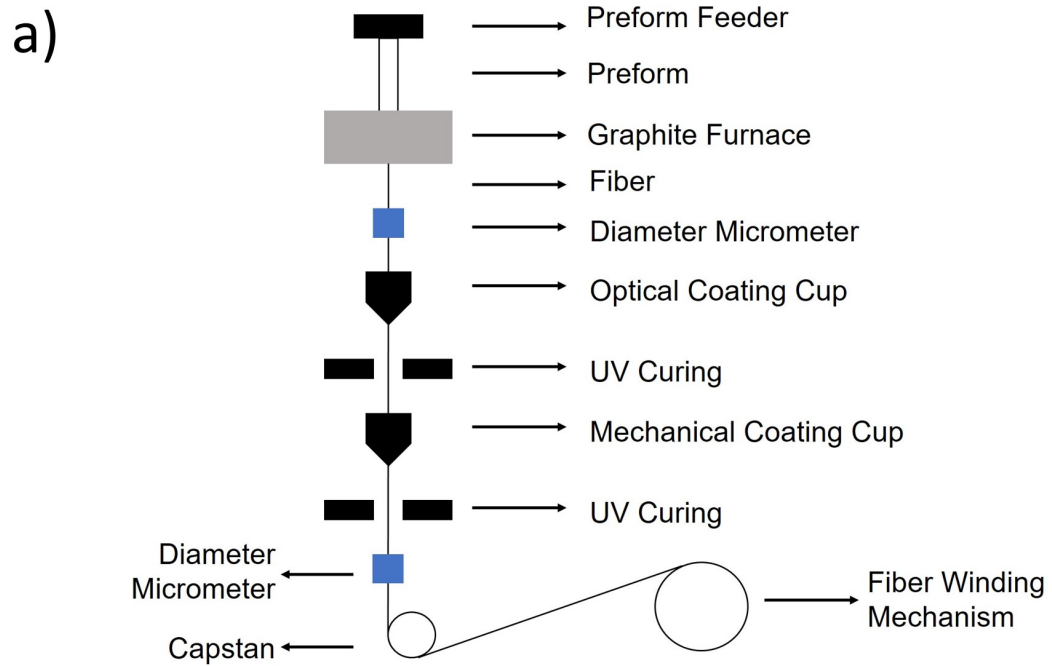


Figure 2.13: a) Fiber drawing tower scheme b) Image of the final fiber shape under optical microscope [23]

2.6 Radiation Effects in Optical Fibers

Exposing optical fibers to ionizing radiation (Gamma-ray, X-ray) will lead to the increased absorption and decreased transmission levels over certain wavelengths. This increased absorption depends on the composition of the optical fiber, the concentration of the elements doped into the fiber, fiber structure and geometry, fabrication conditions as well as the radiation's dose rate, total dose, energy, and ambient temperature. [15, 57–59] Apart from the defects created during fabrication, radiation forms additional defects in fiber structure, which causes the light power at the fiber output to decrease by absorbing the light through the fiber. These defects include intrinsic and extrinsic defects caused by the silica and the elements doped into fiber, respectively. These defects are called color centers and the transmission loss due to radiation is named radiation induced attenuation (RIA). [60–64]

2.6.1 Radiation Induced Attenuation (RIA)

Due to the fact that defects with absorption bands are created when the fiber is exposed to radiation, the attenuation increases and the transmission efficiency of the fiber decreases, which reduces the output light power. It is the measure of the increased loss due to the radiation and is called RIA. [15] It can be calculated as:

$$RIA(\lambda) = -\frac{10}{L} \log \frac{I(\lambda)}{I_0(\lambda)} (dB/m) \quad (2.23)$$

where L is the fiber length, λ is the wavelength of the light, $I_0(\lambda)$ and $I(\lambda)$ are the light intensity before and after the radiation exposure. [65] RIA is dependent on the radiation level, light wavelength, fiber composition, time, and temperature. Most of the color centers created have their absorption bands in UV and visible wavelengths of the spectrum. Therefore, RIA at these ranges is higher than the rest of the spectrum. [61] Doping elements like Al, P, and REs lead to the formation of additional light absorbing defects; hence, fibers containing such elements have generally higher RIA. [66] Some of these defects can exist over a short period

of time or within a narrow temperature range. Thus, the fiber might partially be recovered from the RIA effect if enough time has passed after the radiation exposure or if the fiber is thermally treated by increasing the ambient temperature over a certain degree. [13,67,68] The severity of ionizing radiation effect in optical fibers can also be decreased by adjusting the fiber composition [69], treating the fiber with high energy photons [70], called Photobleaching (PB), and pretreating the fiber before the radiation exposure [71,72].

When the amount of gamma radiation exposure increases, the color centers form and grow. This growth of the color centers can be calculated from the “stretched” second-order growth kinetics. [73]

$$N[(kt)^\beta] = N_{sat} \tanh[(kt)^\beta] \quad (2.24)$$

where N_{sat} is the saturation population of color centers, k is the effective rate constant, β is a number ($0 < \beta < 1$) and t is time which is calculated as Dose/Dose Rate. The dose rate in this study was 1.19 kGy/h.

2.6.2 Color Centers

Color centers are defect sites within the structure of crystalline materials that introduce additional absorption and emission bands. They might occur by a negative ion missing from a particular position; thus, creating a hole filled by electrons trapped by positive ions. Moreover, they may also form by the movement of ions to unusual lattice positions. In addition to their formation during fabrication, color centers can also be created by exposure to ionizing radiation like gamma rays or high-energy photons like laser beams which lead to radiodarkening (RD) and photodarkening (PD), respectively. These powerful, high-energy rays or photons are strong enough to create electrons and holes or misplace ions to create the defect sites.

Color centers are responsible for the RIA in optical fibers. They may be stem from base material SiO_2 and from doped elements. Figure 2.14.a. shows the ideal silica structure in which each Si atom bonds with four O atoms. Each O atom acts

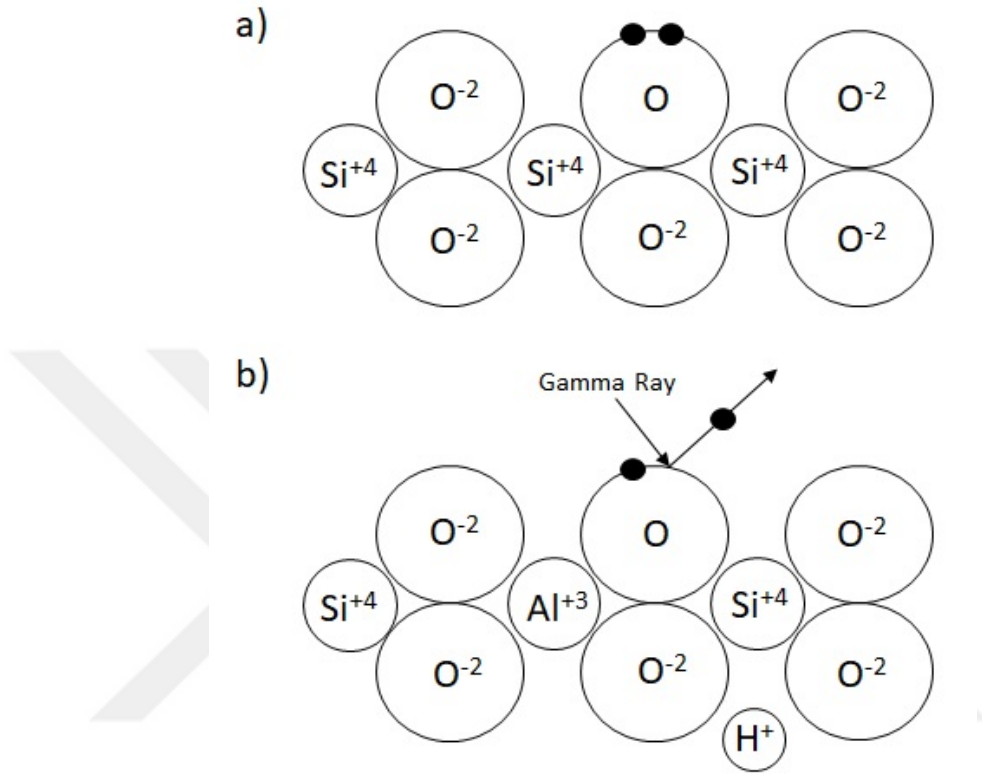


Figure 2.14: a) Ideal SiO_2 structure b) Hole creation after radiation exposure

like a bridge between two Si atoms. In Figure 2.14.b., Al-doped SiO_2 structure can be seen. When the Al atom replaces a Si atom, a +1 valence like hydrogen (H) atom or sodium (Na) ion bonds to the O that could not make enough bonds to satisfy the electroneutrality laws. This condition weakens the electron bonding strength of the O atom. Thus, high-energy gamma rays or photons can pull off an electron, creating a hole named a hole type color center. Similarly, an electron color center is realized when a negative ion is replaced with an electron in the ionic crystalline structure by the effect of radiation.

Color centers absorb the light at specific wavelengths with respect to their electronic structure. The amount of the color centers, as well as the wavelength of the absorbed light, depend on the radiation type, fiber structure and the elements doped into the fiber. [61] The defects are named intrinsic and extrinsic if they originate from silica structure itself or the doped elements like Al, P, Ge, REs,

respectively. [62, 74, 75]

2.6.2.1 Intrinsic Defects

Intrinsic defects come from the primary building material of optical fibers SiO_2 . The ideal tetrahedron structure of SiO_2 is given in Figure 2.15.a. Deviations from this ideality in terms of oxygen excess or deficiency lead to the formation of intrinsic defects. Some of the main intrinsic defects were given below, but they are not limited to them.

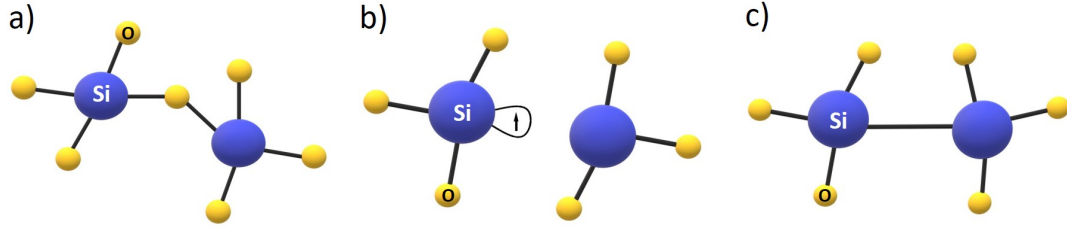


Figure 2.15: a) Ideal SiO_2 structure b) E' center c) Oxygen Deficiency Center (ODC) models

E' center ($\equiv\text{Si}\bullet$) forms when one of the four oxygen atoms is missing and leaving an unpaired electron behind (Figure 2.15.b.). It is the most often studied paramagnetic intrinsic defect of the silica structure. Its structure is well understood by many different Electron Paramagnetic Spectroscopy (EPR) studies. It has an optical absorption (OA) band at 215 nm [74, 76], and has no Photoluminescence (PL) bands.

Oxygen Deficient Center (ODC) (Figure 2.15.c.) is a diamagnetic intrinsic defect and divided into two variations. ODC(I) ($\equiv\text{Si}-\text{Si}\equiv$) consists of a missing O atom bridging between two Si atoms and it has an OA band at 163 nm. ODC(II) ($=\text{Si}:$) is a twofold Si with two unpaired electrons. It has OA bands at 395, 250, and 180 nm and PL bands peaking at 460 and 282 nm. [62, 74]

The first intrinsic defect that is caused by oxygen excess is the *non-bridging oxygen hole center* (NBOHC, $\equiv\text{Si}-\text{O}\bullet$). It is a paramagnetic defect occurring

when there is an O dangling bond where that O atom is bonded to Si (Figure 2.16.a.). NBOHC has OA bands at 620, 260, and 185 nm and a PL band at 650 nm. [74, 77, 78] Its EPR measurement is done at low temperatures to distinguish its signal from other defects.

Peroxy radical (POR, $\equiv\text{Si-O-O}\bullet$) is another paramagnetic oxygen excess type defect in silica. It contains an O atom with an unpaired electron is bonded to another O atom (Figure 2.16.b.). It has OA bands peaking at 620, 260, and 250 nm with no PL band. [78–80]

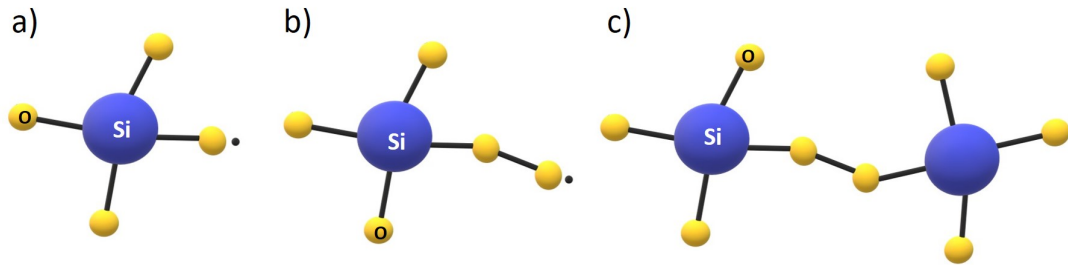


Figure 2.16: a) NBOHC b) POR c) POL models

Peroxy linkage (POL, $\equiv\text{Si-O-O-Si}\equiv$) is a diamagnetic defect occurring when the Si atom between two O atoms is missing (Figure 2.16.c.). Its OA bands peak at 325, 295, 170 and 165 nm. [81–83]

Self-Trapped Holes (STH) are one of the paramagnetic intrinsic defects of silica structure and they are divided into two groups. STH_1 contains a hole trapped on a normal bridging O whereas STH_2 comprises a hole trapped on two normal oxygens (Figure 2.17.a. and b.). The former has OA bands at 475 and 660 nm and is stable below 180 K, and the latter has OA bands at 575 and 760 nm and is stable below 140 K. [84–86]

The OA bands of Si-related intrinsic defects mentioned above and some others can be seen from Figure 2.18.

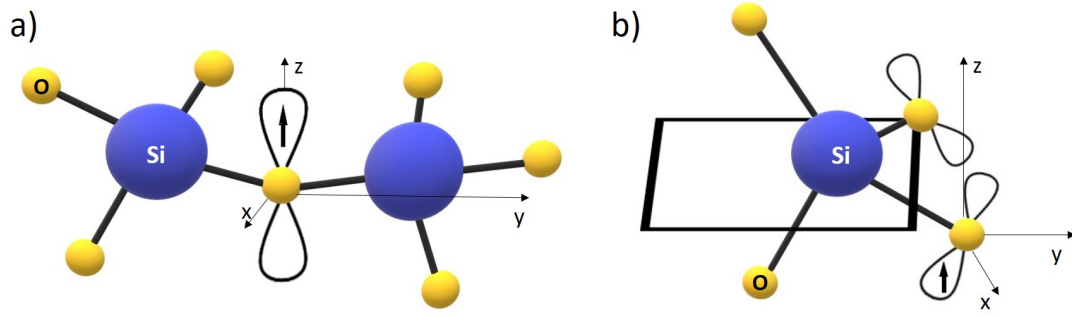


Figure 2.17: a) STH₁ and b) STH₂ models

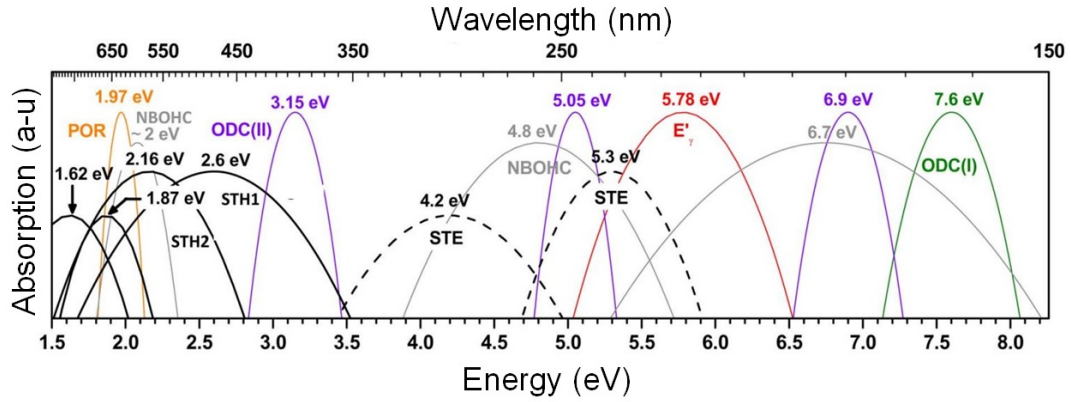


Figure 2.18: OA bands of Si-related intrinsic defects [15]

2.6.2.2 Extrinsic Defects

Extrinsic defects form due to the existence of doped elements in optical fibers. These elements might include Al, P, Ge, REs as well as impurities like H.

H reveals itself as OH⁻ group within the fiber structure. The main effects of the OH⁻ groups are the OA bands peaking at 880, 1140, 1250, 1380 nm. [87] Even though these OA bands deteriorate the transmission abilities of the fiber, H₂ loading and D₂ treatments of the fibers can be utilized to terminate the effects of radiation induced defects in some conditions.

P is an element commonly doped into the core and the cladding of fibers in P₂O₅ form. In addition to the fact that P₂O₅ increases the RI of silica (Figure 2.4.), it decreases the melting temperature of silica; hence, it allows the easier

processing. Furthermore, it decreases the viscosity of glass. [88] Another reason why P is codoped with RE ions is that RE ions have low solubility in silica and form clusters if they are doped above that solubility limit, damaging fiber's light guiding skills. P prevents RE ions from forming clusters and enhances the light transmission abilities of the fiber. [89] P acts as a shield that separates the optically active RE ions [13] and reduces the probability of cooperative energy transfer between them, which might lead to hazardous concentration quenching in which an excited state energy moves between the ions until it finds an appropriate place to decay non-radiatively resulting in photon loss. Another concern with the clustering is “pair-induced quenching” where a pair of Er ions at $^4I_{13/2}$ state combine their energies to move one ion to a higher energy level $^4I_{11/2}$ while the other goes to ground level. Decaying back to the $^4I_{13/2}$ state is non-radiative, which also causes photon loss. Prevention of clustering is quite important because it results in the reduction of the luminescence lifetime of RE, which causes the gain efficiency to decrease. [56] However, the addition of P leads to the creation of P-related defects when the glass is exposed to radiation. Figure 2.19. shows the structural models for some of the main P-defects.

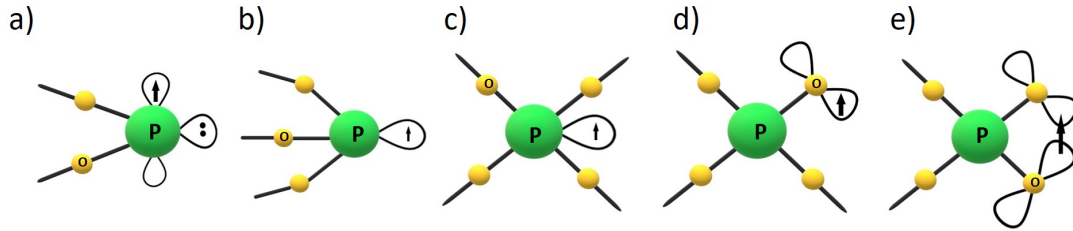


Figure 2.19: a) P_4 b) P_1 c) P_2 d) $POHC^m$ and e) $POHC^s$ models [63]

The five main P-defects are all paramagnetic and their structures are well understood by EPR measurements. [63]

P_1 , P_2 and P_4 are defects where P atom is bonded to 3, 4 and 2 O atoms, respectively. Their OA bands peak at 1570, 275, and 260 nm, respectively. [63]

Phosphorus oxygen hole center (POHC) has two variations depending on its stability. Metastable POHC ($POHC^m$) has an unpaired electron located on a nonbridging oxygen (Figure 2.19.d.) and its OA bands are at 565 and 496 nm.

The stable POHC (POHC^s), however, has two nonbridging oxygens sharing the unpaired electron (Figure 2.19.e.) and its OA bands peak at 235 and 400 nm. [63] When it was first discovered, it was thought that POHC^m was not stable at room temperature. However, DiFrancesca *et al.* found out that the EPR signal of POHC^m was still detectable up until 190°C but not at 280°C. [90]

P_1 might be the most important among all these P-defects since its OA band lies on NIR part of the spectrum covering the telecommunication band on which a lot of application relies on.

OA bands of the P-defects mentioned above can be seen from Figure 2.20.

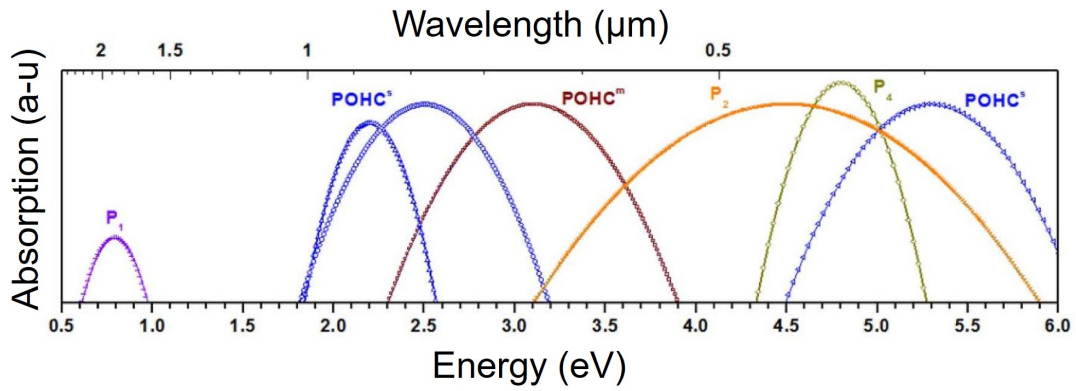


Figure 2.20: OA bands of some of the P-defects [15]

Al is another element that is frequently incorporated into optical fiber core in the form of Al_2O_3 , which increases the RI of silica. The most important mission of Al in optical fibers is to increase the solubility of RE ions in silica. It opens up enough space in the silica structure so that RE ions can be deposited. Al also acts as an agent to block the RE clustering, which can be pretty detrimental to fibers' optical properties. Nevertheless, Al's addition leads to the formation of defect precursors and forms color centers eventually when the fiber is exposed to radiation.

Aluminum oxygen hole center (AlOHC , $\equiv\text{Al}-\dot{\text{O}}-\text{Al}\equiv$) is a paramagnetic Al-defect where there is a hole on the O atom that is bonded to the Al atom. The OA bands of AlOHC peak at 540, 388, and 253 nm. [91]

Al-E' ($\equiv \text{Al}\bullet$) is another paramagnetic Al-defect with a similar structure to the Si E' center. Its OA band is at 253 nm. [91]

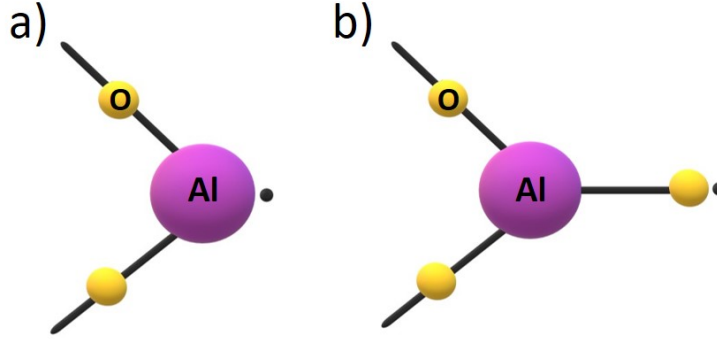


Figure 2.21: a) Al-E' and b) AlOHC models

RE ions are used to obtain the lasing properties in active optical fibers. In this study, Yb-doped optical fibers were used; therefore, the focus was on Yb-related defects. Yb^{+3} ion allows the mentioned lasing properties. However, radiation exposure can decrease the valence of Yb ions from +3 to +2 by electron trapping, which leads to the formation of Yb-related color centers and deteriorates lasing. This transformation takes place as



and is called charge transfer (CT). Excited energy transfer from Yb^{+3} to Yb^{+2} result in a non-radiative decay at the defect site and loss of a photon. The proposed model for this process can be seen in Figure 2.22. The creation of Yb^{+2} creates additional OA bands and leads to RIA and PD, which are crucial concepts for Yb-doped optical fibers. Many studies investigated the CT and Yb^{+2} OA bands were determined as 230 nm [92], 192 nm [93], 210 nm [94] and 330 nm [95].

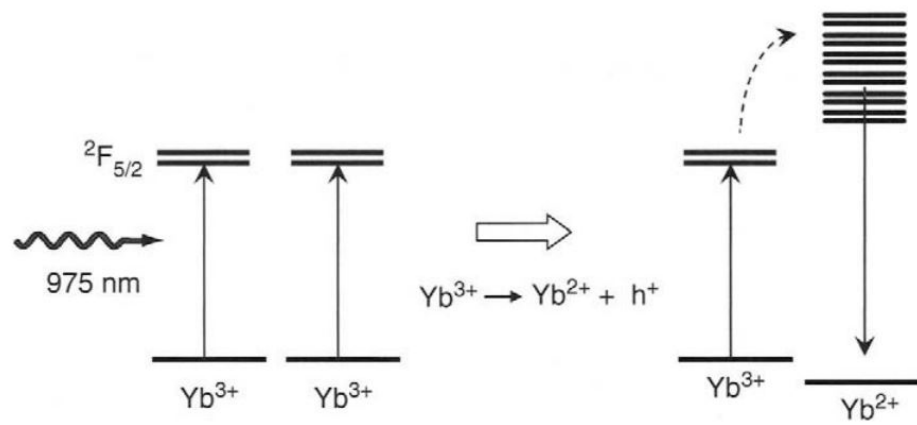


Figure 2.22: Proposed model for the energy transfer from Yb^{+3} to Yb^{+2} [57]

Chapter 3

Experiments

3.1 Preform Characterizations

3.1.1 Polariscopes

Three different preforms were produced for this study, namely Preform 1, 2, and 3. Then, these preforms were drawn into fiber to form the samples Fiber 1, 2, and 3, respectively. After their fabrication is completed, the characterization of the optical preforms starts with a visual inspection method by using the polariscopes, which enables to observe the core and the cladding by using polarized light and taking advantage of the difference between their RIs. The uniformity of the cladding and the core, as well as the presence of bubbles, can be identified. Figure 3.1. shows the polariscopes and the visual inspection that reveals the contrast between the fiber core and the cladding.

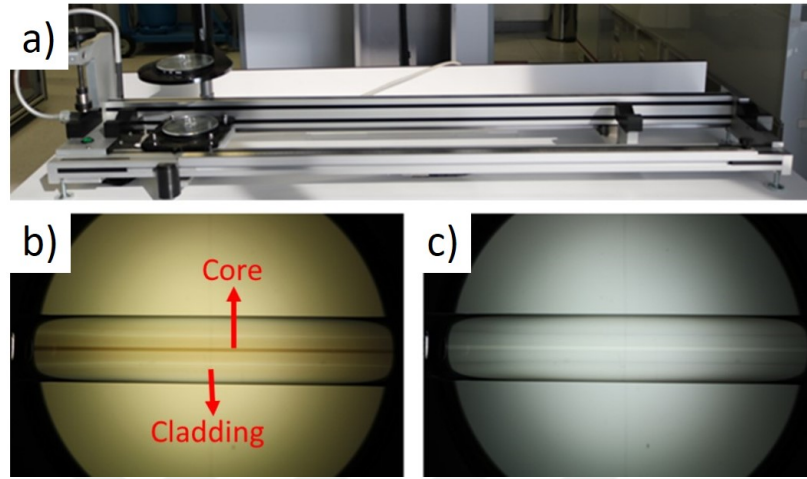


Figure 3.1: a)Polariscope b,c) Inspection of an optical preform with different light polarizations

3.1.2 RIP Analysis

RIP of an optical preform is constructed with York P102 Preform Analyzer, giving results at different angles along the preform length. It measures the RIP by shining a 633 nm light on the preform immersed in index matching oil and collecting the scattered light. Different elements result in different scatterings and the software uses this data to plot the RIP. Figure 3.2. shows the mentioned preform analyzer.



Figure 3.2: Preform analyzer is used to obtain RIP profile of the preform

3.1.3 Analysis of Yb concentration

The extent of Yb doping into the optical preform is conducted by the Measurement of Optical Preform Slice (MOPS). A slice of the preform is cut and polished and the MOPS gives the Yb^{+3} mol% in the preform by measuring the amount of light absorbed by the Yb^{+3} ions and converts this data into Yb^{+3} mol%. The lower the light intensity, the higher the Yb^{+3} mol%. MOPS uses the formula given below to perform this measurement.

$$\alpha = \frac{1000}{L} 10 \log \frac{P_{core}}{P_{baseline}} \quad (3.1)$$

Where α is the light absorption coefficient, L is the slice thickness, P_{core} and $P_{baseline}$ are the light intensities at the core and at the cladding, respectively.

3.2 Fiber Characterizations

3.2.1 Elemental Analysis

Which elements present in fibers and up to what extent are two very crucial questions in optical fiber production. The compositional analysis of fibers is made with a Wavelength-dispersive X-ray Spectroscopy (WDS). WDS is very similar to Energy-dispersive X-ray spectroscopy (EDS). However, the former evaluates the wavelengths of X-rays emitted by the elements one by one, whereas the latter checks the energies of X-rays emitted by elements all at once. Therefore, WDS gives more delicate and improved measurements than EDS, which is its most significant advantage over EDS.

3.2.2 Geometric Analysis

Geometric analysis is conducted to find out about the octagonal shape (ratio of side diameter to corner diameter) as well as to see if the fiber is centered after

the coating process. All the fiber samples have 20 μm core and 400 μm cladding diameters. (Figure 2.13.b.)

3.3 Light Intensity Measurements of Pristine Fibers

A 100 W ozone-free xenon lamp (Oriel LCS-100) was used to measure the intensity of the transmitted light by the fiber samples over the range from 300 to 1100 nm. Each fiber sample was 2 meters long. Light coming from the Xe lamp is first coupled into a quartz fiber using some optical apparatus like lenses and irises to prevent the optical spectrometer (Ocean Optics HR4000CG-UV-NIR) from saturating by decreasing the light intensity. Then, this quartz fiber is connected to the fiber samples via a connector. Finally, the fiber samples are connected to the spectrometer and the intensity spectrums are recorded by OceanView software. The scheme of the optical system is given in Figure 3.3.

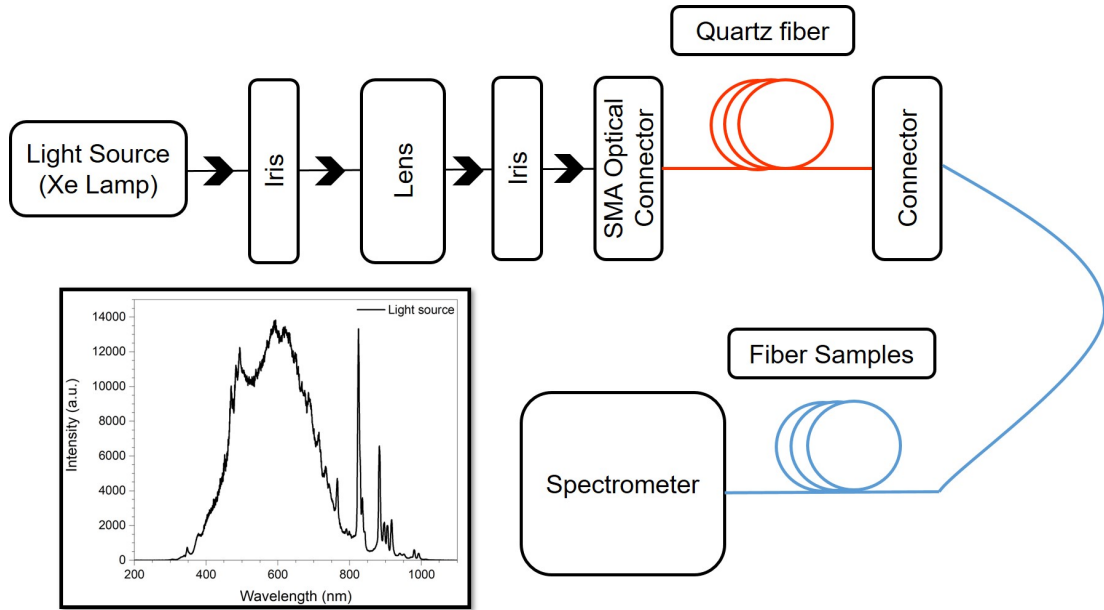


Figure 3.3: Optical setup for the intensity spectrum measurements. The inset shows the intensity spectrum of the Xe lamp.

3.4 Gamma-Ray Irradiation of Fibers

Fibers are exposed to gamma-rays with a Co-60 gamma radiation source in a pool. The irradiation setup can be seen below. The dose rate was 1.19 kGy/h and the total doses were 0.5, 1, 10 and 50 kGy. The irradiation setup is shown in Figure 3.4.

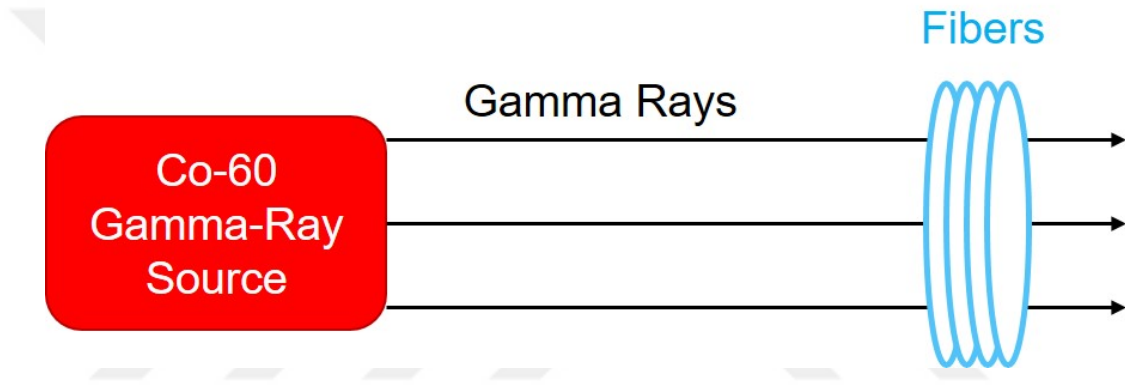
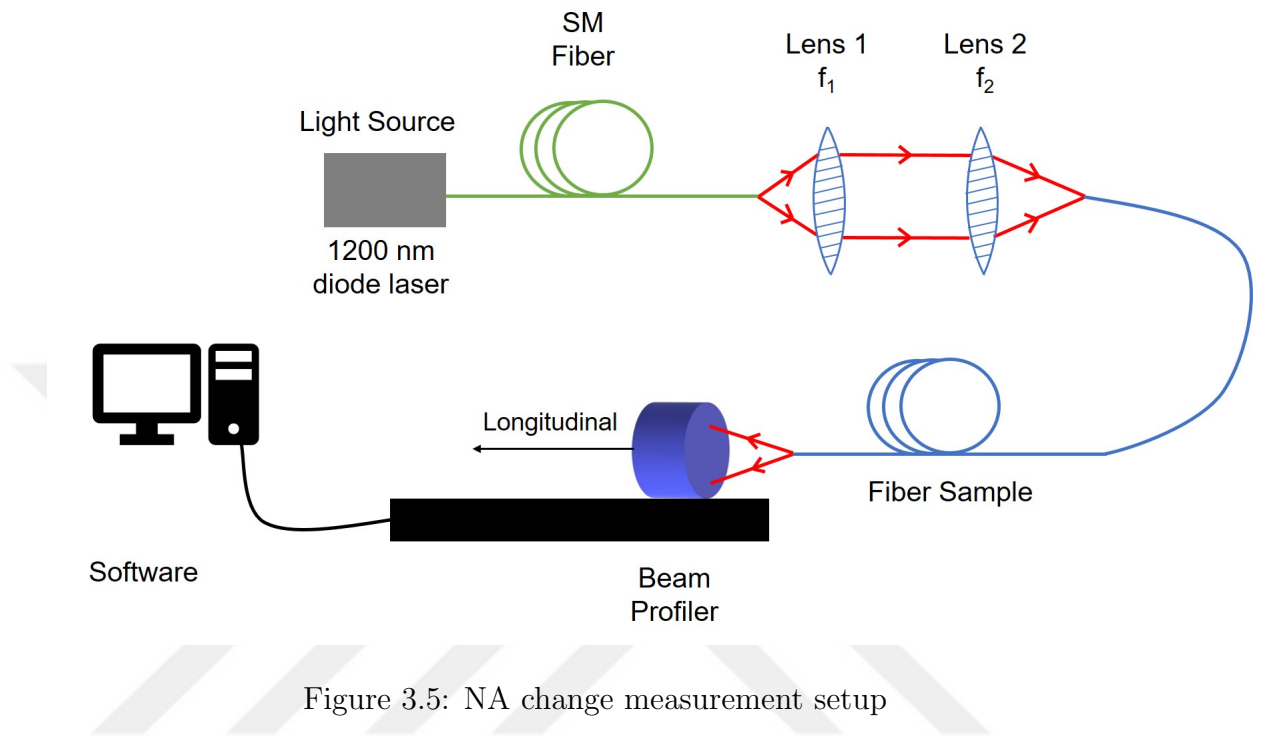


Figure 3.4: Irradiation of fibers with gamma radiation

After the radiation exposure, light intensity spectra of the irradiated fibers were measured again in the same optical setup as Figure 3.3. Then, the transmission losses and the RIA was determined.

3.5 NA Measurements

The effects of gamma radiation on Yb-doped optical fibers were also observed in NA measurements. The NA measurement setup is given in Figure 3.5. 1200 nm diode laser was used as the light source coupled into an SM fiber, after which the output light was first collimated and then coupled into the fiber samples by using two lenses. Finally, the light coming out from fiber samples was analyzed by THORLABS BP109-IR - Slit Scanning Beam Profiler, which was mounted on MICOS POLLUX DRIVE RS-232 that enabled the beam profiler to move longitudinally.



Chapter 4

Results and Discussion

4.1 Characterization Results

4.1.1 RIP Analysis

Figure 4.1. reveals an exemplary RIP of the optical Preform 2 used in this study and it shows the RI difference between the core and the cladding of Preform 2. The reason for the dips in the RIP around 1.2 mm diameter is that fluorine incorporation into the inner walls of the substrate tube occurred during the tube's cleaning with SF_6 . The core that lies at the center of the preform has a higher RI than the cladding due to the deposition of elements like Yb, Al, and P into this region, increasing the RI of silica. The RIP variations within the preform core might have occurred due to the evaporation and re-incorporation of P_2O_5 during the high-temperature preform collapsing stage.

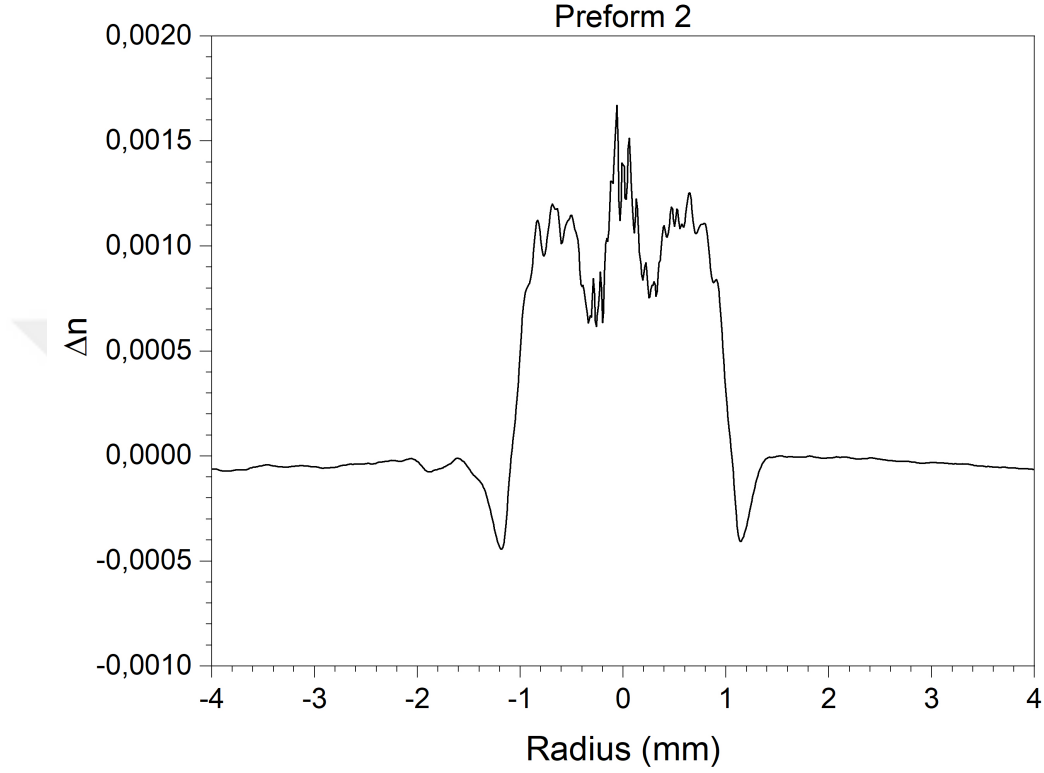


Figure 4.1: RIP of the optical Preform 2

4.1.2 Analysis of Yb concentration

The amounts of Yb^{+3} mol% obtained from MOPS are given in Table 4.1. The measurement for Preform 4 could not be performed simply because that commercial sample was purchased in fiber form, not in the preform. An exemplary MOPS graph of Preform 2 is given in Figure 4.2.

Table 4.1: Yb^{+3} mol% of preform samples

	Preform 1	Preform 2	Preform 3
Yb^{+3} mol%	0.03	0.09	0.12

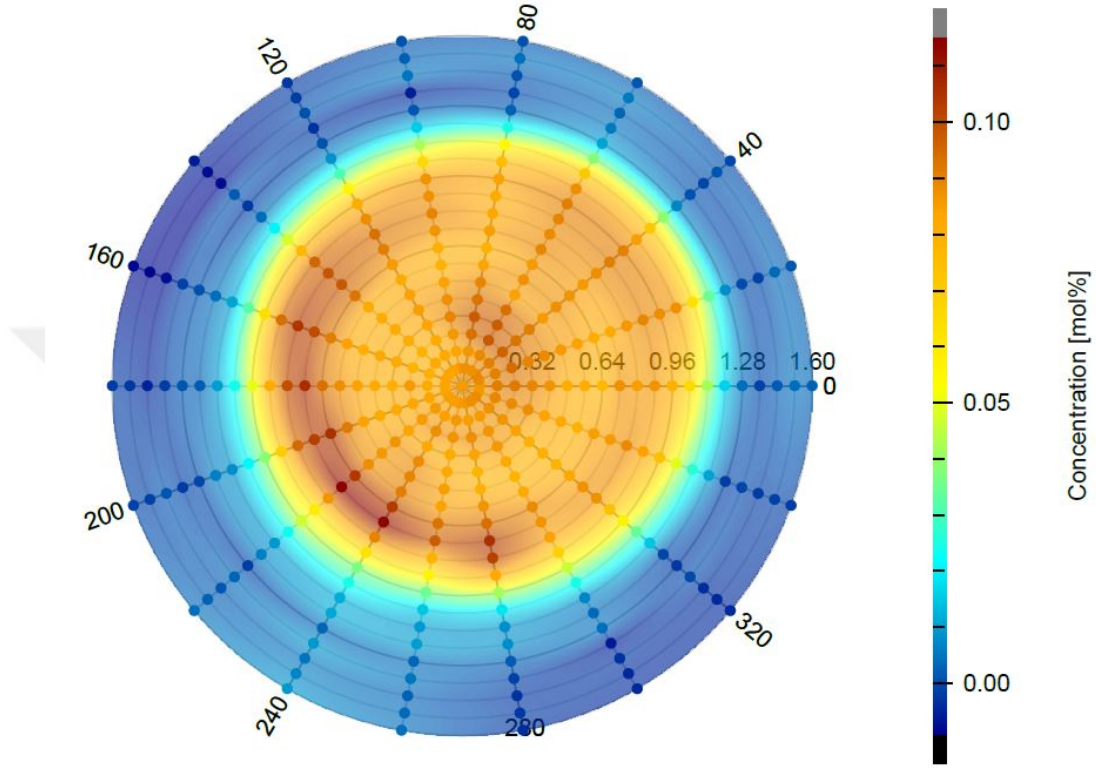


Figure 4.2: MOPS graph of Preform 2

Orange and red regions in Figure 4.2. implies that the light absorption by the Yb^{3+} ions is higher, whereas the blue regions mean less Yb^{3+} absorption. Therefore, the density of Yb^{3+} ions in orange and red regions is higher than blue.

4.1.3 Elemental Analysis of Fibers

The compositional analysis of the fiber samples used in this study can be seen in Table 4.2. Fiber 4 is commercially available Nufern LMA-YDF-20/400, whereas the rest of the fibers are home-made. The results for the Yb ions are pretty close to those obtained from MOPS; however, the differences are caused by the fact WDS measures all Yb atoms, including Yb^{+3} and Yb^{+2} whereas MOPS measures only Yb^{+3} .

Table 4.2: Elemental analysis of the four fiber samples

Mol %	Fiber 1	Fiber 2	Fiber 3	Fiber 4
Yb_2O_3	0.03	0.14	0.15	0.178
Al_2O_3	1.80	1.04	1.81	1.308
P_2O_5	2.07	1.28	1.82	1.423
AlPO_4	3.60	2.08	3.62	2.616
P/Al	1.15	1.23	1	1.09
Al/Yb	60	7.4	12.1	7.3

4.2 Measurements of the Transmission Losses and the RIA

The light intensity spectra of pristine fiber samples and immediately after irradiation are given in Figure 4.3.a-d. It can be seen that the light intensity decreases and the RIA increases with increasing radiation dose, which proves the presence of RIA, respectively. RIA was calculated from this intensity data by the formula (2.23) and was shown in Figure 4.3.e-h. It is evident that the transmission losses are much more severe over the visible range than over NIR due to the fact that the absorption bands of most of the color centers reside over UV and visible ranges.

Fiber 1's light transmission abilities had deteriorated significantly when exposed to 0.5 kGy gamma radiation, as shown in Figure 4.3.a. but increasing the radiation to 1 kGy made only a slight difference. A greater transmission loss was induced when the radiation level reached 10 kGy causing further damage. However, exposing Fiber 1 to much heavier 50 kGy radiation could only cause additional transmission loss at wavelengths greater than 600 nm. Therefore, it can be inferred that 1 kGy radiation created color centers slightly more than 0.5 kGy. In contrast, there must be significant amount of new color center formation when going from 1 kGy to 10 kGy. The transition from 10 kGy to 50 kGy must have created color centers whose OA bands are beyond 600 nm. The RIA spectrum of Fiber 1 in Figure 4.3.e. agrees with the assumptions made above. There is barely any difference between the attenuation levels of 0.5 kGy and 1 kGy. Following a significant increase in RIA from 1 kGy to 10 kGy, there is a higher raise in attenuation levels at 50 kGy after ~ 600 nm reaching 6 dB/m.

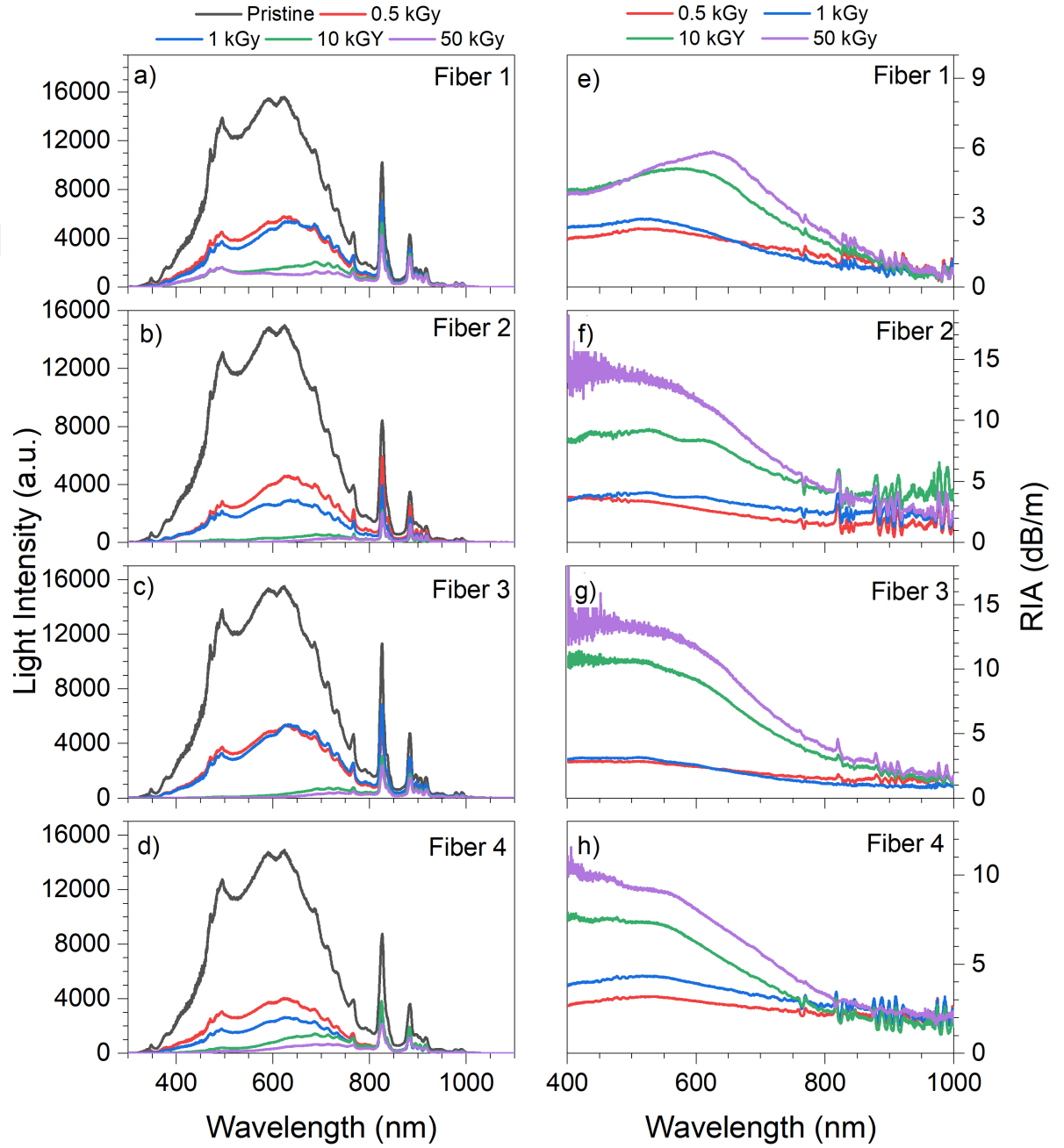


Figure 4.3: a-d) Light intensity spectra for Fiber 1, 2, 3, 4 and e-h) RIA spectra for Fiber 1, 2, 3, 4 obtained from intensity spectra

Fiber 2 experienced significant radiation induced loss at 0.5 kGy gamma radiation, especially at UV and visible ranges, as shown in Figure 4.3.b. Raising the radiation to 1 kGy did not cause additional loss up to ~ 500 nm; however, the transmission loss increased at greater wavelengths. At 10 kGy, the transmission ability of Fiber 2 was affected immensely that decreased the light intensity to very low values. The radiation induced loss has increased even further at 50 kGy so that the intensity spectrum is hardly seen below ~ 700 nm. The RIA spectrum of Fiber 2 in Figure 4.3.f. shows similar RIA levels for 0.5 kGy and 1 kGy. However, RIA increases significantly for both 10 kGy and 50 kGy reaching 15 dB/m at ~ 450 nm. The attenuation spectrum decreases at higher wavelengths but still reaches to NIR region.

Fiber 3's behavior under gamma radiation was similar to that of Fiber 2. As shown in Figure 4.3.c., a high transmission loss at 0.5 kGy was followed with almost no change at 1 kGy. However, 10 kGy and 50 kGy gamma radiation deteriorated the light transmission abilities of Fiber 3 considerably so that the light intensity spectra almost disappeared below 700 nm, which induced large losses. This radiation induced loss can be seen better in Figure 4.3.g. Having started with lower attenuation levels, RIA reached 15 dB/m at ~ 450 nm at 50 kGy and it decreased at higher wavelengths as expected.

Fiber 1, 2, and 3 showed similar spectra in terms of similar RIA for 0.5 kGy and 1 kGy followed by a big jump from 1 to 10 kGy and reaching even further losses at 50 kGy. The RIA is mainly caused by the hole centers related to Al and P and some intrinsic defects. These defects leading to the formation of color centers must have been created at a much higher rate at 10 kGy than 1 kGy. The similarity between the 0.5 kGy and 1 kGy RIA spectra might be due to the fact that 1 kGy radiation simply was not strong enough to create additional color centers as 10 kGy and 50 kGy did. Relatively higher P_2O_5 mol%, as well as P/Al ratios of these samples, might be the cause of their similar behavior under gamma radiation effect.

Fiber 4 showed a somewhat gradual decrease in the light intensity spectrum as the radiation increases. 0.5 kGy and 1 kGy gamma radiation caused significant

induced losses. Moreover, 10 kGy and 50 kGy radiation increased this loss even further. However, The latter radiation levels affected the wavelengths below ~ 600 nm much more severely. Figure 4.3.h. agrees with these results as the RIA reaches 10 dB/m near 400 nm and is higher than around 600 nm. This might be inferred as the color center formation rate at lower wavelengths was higher than at greater wavelengths. The lower Al/Yb ratio of Fiber 4 can explain such behavior as this was a similar behavior in Fiber 2 and 3 whose Al/Yb ratios were lower than Fiber 1.

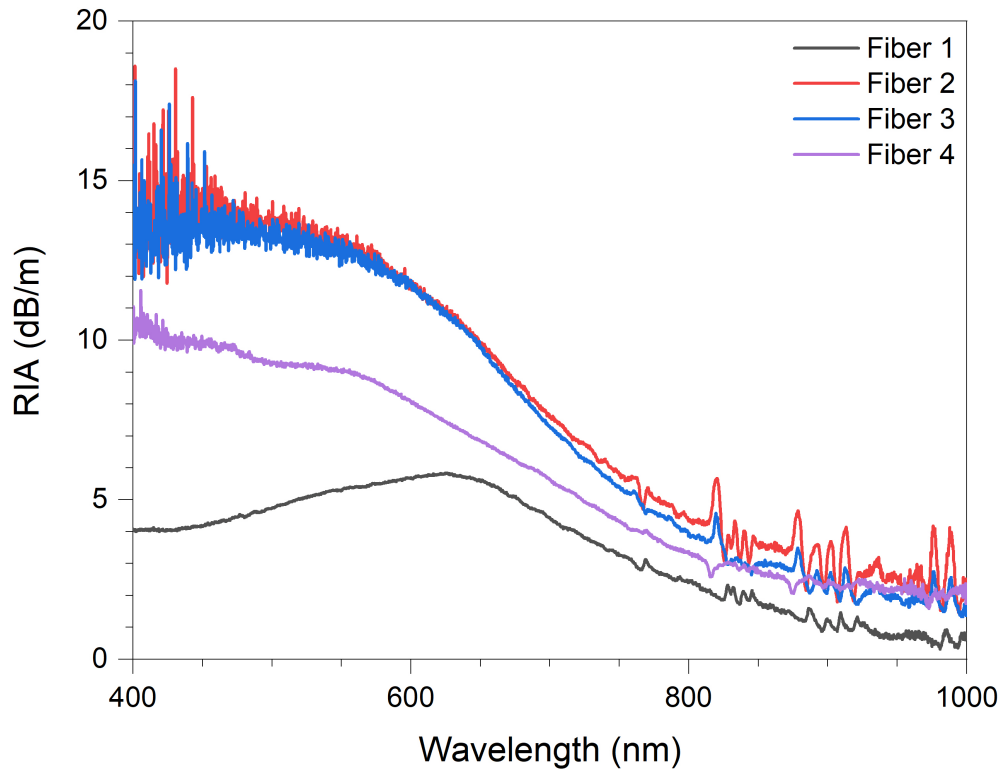


Figure 4.4: Comparison of RIA values of fiber samples at 50 kGy gamma radiation

A comparison of the RIA values of fiber samples after 50 kGy gamma radiation exposure can be seen in Figure 4.4. Interpreting the Table 4.2., Figure 4.3. and Figure 4.4. together, the following discussions can be made. It can be inferred that Fiber 1 was the least affected sample by the radiation since its RIA values are the lowest. Fiber 4 was less resistant to gamma radiation than Fiber 1, where Fiber 2 and Fiber 3 were the most severely affected samples. The variations in

radiation hardness are the result of the differences in the sample compositions. The main reason why Fiber 1 was affected the least is that it has the lowest Yb mol% doped in its core. The amount of Yb in Yb-doped fibers acts as the most critical RIA sensitivity parameter. Furthermore, Fiber 1 has an Al/Yb ratio of 60, which is the highest among the samples and it could have helped to better radiation resistance. Higher Al/Yb ratio is beneficial for more homogenous Yb doping due to the fact that Al prevents the Yb clustering from occurring. In addition, Fiber 1 has a high AlPO_4 mol% formation, which happens when Al and P are doped into fiber core at similar moles. It has been found that the radiation resistance increases with the AlPO_4 amount. [96] Therefore, a high amount of AlPO_4 must have played a significant role in the low RIA levels of Fiber 1.

The rest of the samples contain much higher Yb mol% doping levels; therefore, they all have higher RIA values than Fiber 1. Fiber 4 has the highest amount of Yb doping and it might be expected that it is the most vulnerable sample to radiation damage. However, its P/Al ratio close to that of Fiber 1 and a decent amount of AlPO_4 mol% made Fiber 4 have better radiation resistance and fewer RIA values than expected.

Fiber 2 and 3 were the most severely affected samples and their RIA values were the highest. Fiber 2 has the lowest doping levels of Al_2O_3 and P_2O_5 among the samples, which might not have satisfied the Yb solubility expectations and have caused its light guiding properties to be deteriorated by the gamma radiation. Furthermore, it has one of the lowest Al/Yb ratios. Fiber 3 showed weak radiation resistance, although it had a decent amount of Al_2O_3 and P_2O_5 mol%. However, it has relatively lower Al/Yb and P/Al ratios, as well as higher Yb mol%, which must have acted a role in Fiber 3's significant low radiation resistance. Furthermore, a small number of impurity atoms that might have come from the substrate tube or the precursors, as well as H_2 or Cl_2 incorporation, could have contributed to its low radiation resistance. Moreover, defects that occurred during its fabrication, impurities, structure irregularities, bubble formations might have led to Fiber 3's high RIA values.

The characteristic shape of the RIA spectrum of each sample consists of the

OA bands of the color centers that are created by the radiation exposure. These RIA spectra can be deconvoluted into smaller OA spectra of each color center with respect to their wavelengths. In other words, OA spectra of each color center created by the gamma radiation can be combined to construct general RIA spectra. Figure 4.5.a-d. and e-h. show the RIA spectra for 10 kGy and 50 kGy total doses, respectively, for each sample as well as the color centers forming them up. Figure 4.5.i-l. shows the intensity change of the OA bands of color centers occurred when the total dose increased from 10 kGy to 50 kGy. It can be understood that the increase in the intensities of OA bands for most of the color centers is the reason behind the fact that higher radiation levels cause higher RIA.

Figure 4.5.a-d. and e-h. clearly show that the Al and P doping during the fiber fabrication led to the formation of extrinsic color centers related to these elements, which are ALOHC and POHC. Furthermore, intrinsic color centers like ODC, NBOHC, STH_1 and STH_2 were also formed by the radiation exposure of SiO₂. The color centers created due to gamma radiation might not be limited to the ones shown in Figure 4.5. OA bands of other color centers might reside within the OA bands of the main color centers shown and be shadowed by them. Therefore, they could not be revealed after the deconvolution. It can be seen that the OA band intensity of some of the color centers decreased, although the radiation level increased. This decrease might occur due to the fact that the exposure to more ionizing radiation might have led to the transformation of one color center into another one; hence, decreasing the intensity of the former while increasing that of the latter. Being shadowed by the powerful color centers might be another reason that has led to this intensity decrease.

Color centers due to the Yb ions do not appear in Figure 4.5. due to the fact that their OA bands lay outside of the chosen RIA spectra that is from 400 nm to 1000 nm.

Fiber 1's RIA spectra consist of intrinsic color centers ODC, NBOHC and STH_2 and extrinsic color center ALOHC as shown in Figure 4.5.a.&e. The intensities of these color centers increase as the radiation increases from 10 kGy to 50

kGy except the one that belongs to AlOHC. This decrease does not necessarily mean there is less amount of AlOHC. Contrarily, the population of AlOHC defects increased most probably with the radiation. However, this increase might not have been detected due to how the RIA spectra are deconvoluted. Furthermore, the lack of P-related color centers in Figure 4.5.a.&e. does not mean there are not any. AlOHC intensity decrease and the lack of P-related color centers might have been overshadowed by the OA bands of other color centers.

Figure 4.5.b&f. show the RIA spectra of Fiber 2 at 10 kGy and 50 kGy which include NBOHC, STH₁, STH₂, and AlOHC. It should be taken note that AlOHC has two separate OA bands. Also, the OA band STH₂ was almost invisible for 10 kGy but then increased at 50 kGy. The intensities of each defect increase with the radiation and the lack of P-related defect does not mean there are no color centers derive from P-doping.

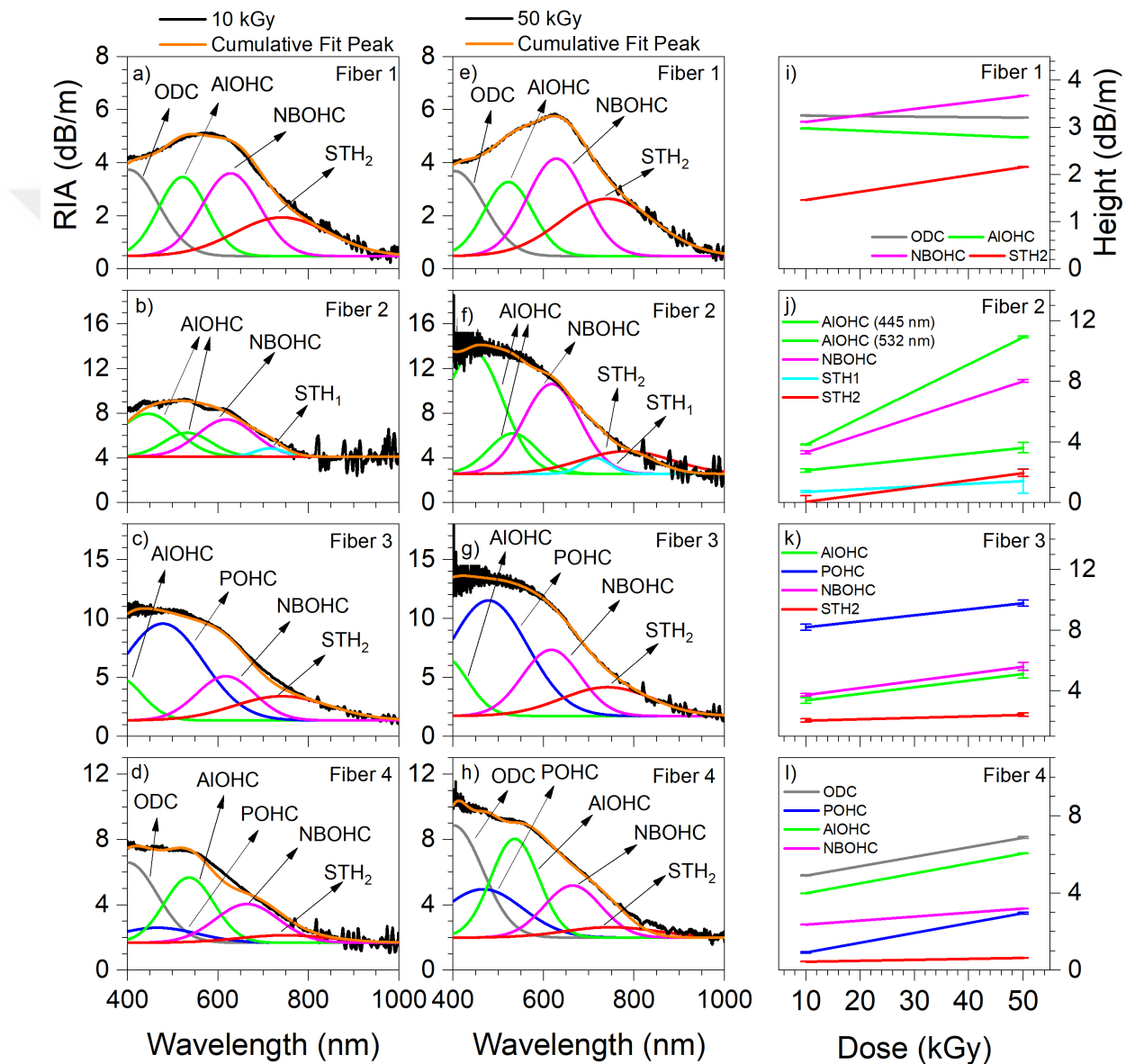


Figure 4.5: a-d) 10 kGy RIA deconvolution for Fiber 1, 2, 3, 4. e-h) 50 kGy RIA deconvolution for Fiber 1, 2, 3, 4 i-l) Intensity changes for the OA bands of color centers between 10 kGy and 50 kGy

Fiber 3's RIA spectra consist of NBOHC, STH₂, ALOHC and POHC, as shown in Figure 4.5.c.&g. Each of the color centers increases its intensity with the radiation, which can be seen in Figure 4.5.k.

Figure 4.5d.&h. show that Fiber 4's RIA spectra consist of ODC, NBOHC, STH₂, POHC and ALOHC. The intensities of the OA bands of each of these color centers increase with the radiation, shown in Figure 4.5.l.

The intensity changes of the OA bands of color centers which were shown in Figure 4.5.i-l. might be interpreted in such a way that a higher intensity increase may mean a higher rate of color center formation when the radiation level increases from 10 kGy to 50 kGy. To illustrate, the rate of ODC formation in Figure 4.5.l. can be higher than that of NBOHC formation. However, this assumption should be considered within the accuracy limits of intensity change measurements.

The formation and the growth of color centers as the amount of radiation exposure increases are described and shown by the stretched second-order growth kinetics, as told earlier. (2.24) The growth kinetics with respect to the radiation dose for fiber samples are shown in Figure 4.6. Five different wavelengths were chosen for this purpose, although any other wavelength within the RIA spectra could be chosen. The flattening of the growth curve as the radiation exposure increases means that the color center population that led to the OA formation at that specific wavelength is saturated. In other words, no more color centers at that wavelength can be created more even if the exposure time or the radiation levels are increased. To illustrate, it seems like more color centers could be created for Fiber 2 whereas the color center population has saturated for Fiber 1.

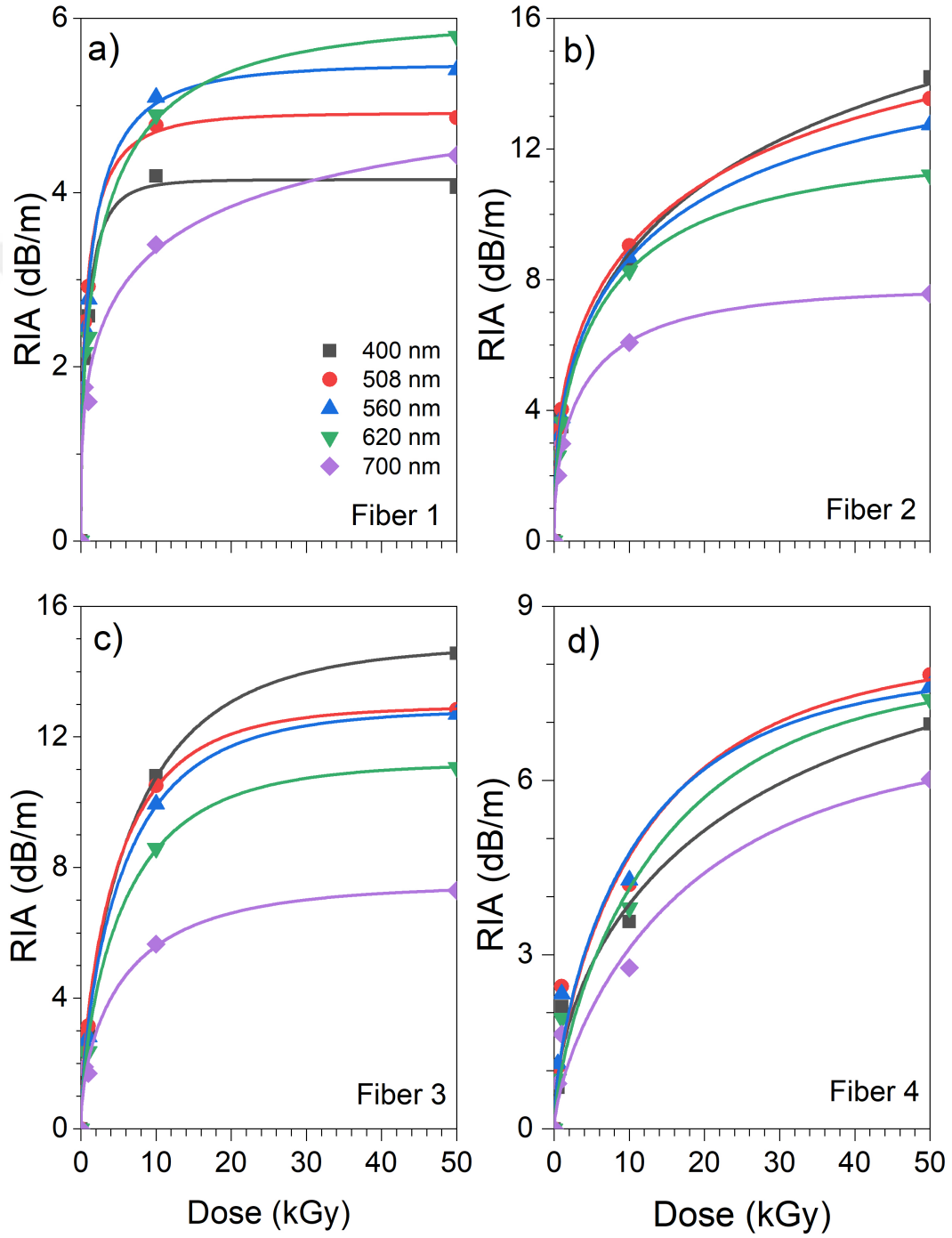


Figure 4.6: Growth kinetics at five different wavelengths for a) Fiber 1 b) Fiber 2 c) Fiber 3 d) Fiber 4

In Figure 4.6.a., it seems like the color centers that absorb light at 400 nm are formed and grow quicker than those at 700 nm for Fiber 1. However, the population of the 400 nm color centers saturates quickly as well. In contrast, the ones at 700 nm continue to increase with the radiation, resulting in higher RIA levels at 700 nm than 400 nm for 50 kGy. This contradicts the assumption that fewer OA absorption bands at greater wavelengths should yield lower RIA, which is the case for all other samples. In Figures 4.6.b-d., it can be seen that the lowest RIA values were obtained for 700 nm.

Fibers 2 and 3, which did not show the contradiction for Fiber 1, exhibited higher RIA at lower wavelengths as expected. Figures 4.6.b.&c. show that the color center population grows and saturates as the radiation increases. It is worth mentioning that Fiber 2 continues to have increasing RIA values at 400 nm and 508 nm, whereas Fiber 3 shows saturation for the same wavelengths. This can be interpreted as further color centers can be created in Fiber 2 at higher radiation levels but not in Fiber 3.

For Fiber 4, although the color center populations saturate at 50 kGy, the fact that the RIA at 400 nm is the second-lowest among all the wavelengths is interesting. It could be expected that RIA should be the highest for 400 nm, which was the case for Fibers 2 and 3. Figures 4.6.d. reveals that less color centers absorbing light at 400 nm were created than those absorbing at 508 nm and 620 nm.

Growth kinetics of color centers are shown for particular wavelengths in Figure 4.7. To illustrate, Fiber 1 has the lowest RIA at 560 nm, as shown in Figure 4.7.c. This means that it has the least amount of color centers created; thus, the most gamma radiation resistant sample at this particular wavelength. Similar graphs can be constructed to find out which sample has the best use at a specific wavelength required by the application.

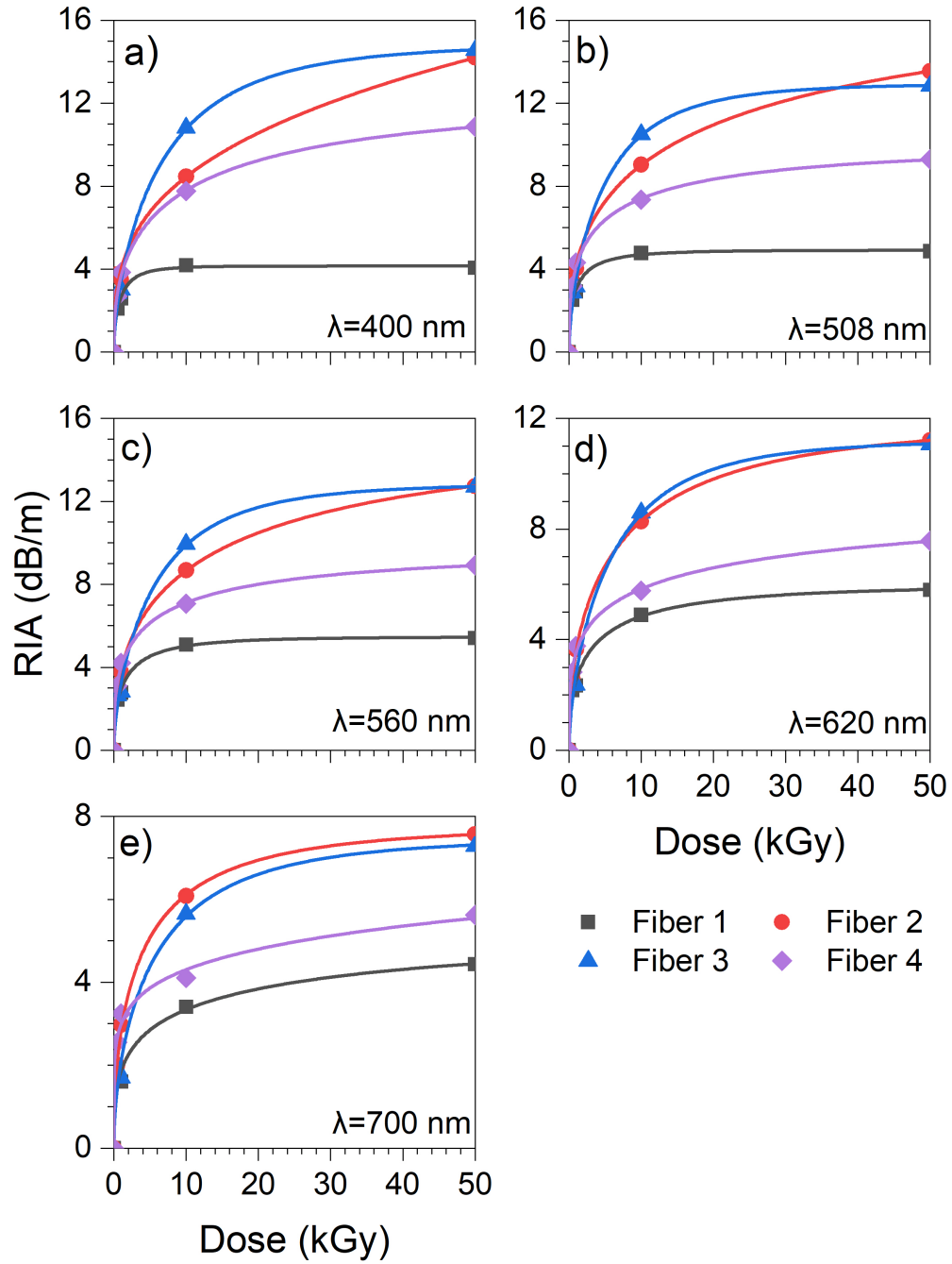


Figure 4.7: Growth kinetics comparison of Fiber 1, 2, 3 and 4 at five different wavelengths for a) 400 nm b) 508 nm c) 560 nm d) 620 nm e) 700 nm

In Figures 4.7.a-d., it can be seen that Fibers 2 and 3 have the highest RIA at all wavelengths, which was already anticipated from Figure 4.4. At all wavelengths, Fiber 1 saturates very quickly to yield lower RIA. Fiber 4 exhibits more radiation hardness than Fibers 2&3. The fact that the highest RIA values that all the fibers can reach decreases with increasing wavelengths agree well with the previous assumptions that claimed fewer OA bands at greater wavelengths.

The fitting parameters for the second-order growth kinetics for the color center growth in optical fibers were given in equation (2.24) and they were shown in Table 4.3. at five different wavelengths for each sample for the dose rate of 1.19 kGy/h. N_{sat} gives the maximum amount of color centers that can be created at that particular wavelength, k is the rate constant for the color center formation and β is a number between 0 and 1.

The N_{sat} tends to decrease as the wavelength increases. This outcome can be expected due to the fact that most majority of the OA bands of color centers lay within the UV and visible regions. Therefore, fewer color centers can be created, which resulted in the decreasing of N_{sat} at greater wavelengths. For the same reason, k values are expected to decrease with increasing wavelength as well since the less amount of color centers results in a lower growth rate constant. β values are predicted to have a value around $\sim 2/3$ and increase with the wavelength. The values in Table 4.3. for N_{sat} , k and β are somewhat comparable to Griscom's work which has explained the behavior of these parameters for optical fibers under the radiation effect. [73] The irregularities in Table 4.3. might have been caused by the small number of data points that are obtained from Figure 4.6. and 4.7. which makes the fitting of equation (2.24) more difficult. This fitting would be more precise and accurate in online RIA measurements where the number of data points is much higher.

Table 4.3: Saturation population, N_{sat} , rate constant, k , and β fitting parameters which were obtained from second-order growth kinetics for samples Fiber 1, 2, 3, 4 and 5

Sample	Wavelength	N_{sat}	k	β
Fiber 1	400 nm	4.15±0.10	0.63±0.17	0.51±0.11
	508 nm	4.91±0.13	0.55±0.13	0.42±0.07
	560 nm	5.47±0.18	0.35±0.10	0.42±0.06
	620 nm	5.98±0.33	0.16±0.06	0.39±0.05
	700 nm	5.35±1.78	0.04±0.09	0.30±0.09
Fiber 2	400 nm	20±0.01	0.016±0	0.38±0.03
	508 nm	17.57±1.58	0.025±0.01	0.37±0.02
	560 nm	15.24±0.77	0.035±0.01	0.39±0.02
	620 nm	11.90±0.14	0.085±0.01	0.44±0.01
	700 nm	7.76±0.21	0.14±0.02	0.44±0.03
Fiber 3	400 nm	14.84±0.63	0.10±0.02	0.59±0.06
	508 nm	12.94±0.49	0.14±0.03	0.60±0.06
	560 nm	12.84±0.53	0.12±0.02	0.60±0.06
	620 nm	11.20±0.54	0.12±0.03	0.59±0.07
	700 nm	7.47±0.58	0.11±0.05	0.53±0.09
Fiber 4	400 nm	12.29±0.72	0.06±0.02	0.38±0.03
	508 nm	10.04±0.58	0.10±0.04	0.33±0.03
	560 nm	9.68±0.60	0.10±0.04	0.33±0.03
	620 nm	9.47±2.00	0.03±0.05	0.26±0.04
	700 nm	9±0.01	0.004±0.002	0.18±0.02

Table 4.3. reveals that Fiber 1 had a color center population increase with the wavelength. Although this contradicts the general view of having fewer OA bands at greater wavelengths, it is compatible with Fiber 1's defect growth graph in Figure 4.6.a. The higher RIA values at around 600 nm can also be interpreted from higher N_{sat} values at 620 nm for Fiber 1 at Table 4.3. The k and β values decrease with increasing wavelength.

Fiber 2 tends to decrease N_{sat} values as the wavelength increases, which agrees well with its defect growth data in Figure 4.6.b. Its k values increase with the wavelength. The significant increase in k parameter when the wavelength increases from 620 nm to 700 nm should be noted. The small number of data points to create a fit as well as the uncertainties in RIA measurement might have created such a big difference. β values of Fiber 2 increase just a bit with the

wavelength.

Fiber 3, which showed a similar RIA behavior to Fiber 2, has a similar trendline in N_{sat} values, decreasing with increasing wavelength. k and β values of Fiber 3 almost are not affected by the wavelength change.

Fiber 4 shows a decrease in its N_{sat} values with increasing wavelength. Moreover, k values exhibit a slight increase first and then a decrease. There is almost a decrease of one order of magnitude when the wavelength increases from 620 nm to 700 nm, which can be caused by the same reasons that of Fiber 2. β values of Fiber 4 experience a decrease with increasing wavelength.

As mentioned in Chapter 3, some of the color centers can be mitigated by temperature or light treatment. The fiber samples were kept at room temperature (RT) after irradiation. Their intensity and RIA spectra were measured for three subsequent weeks to find out how much the fibers recovered from the radiation effect. The recovery spectra were also compared with the spectrum measured right after irradiation to visualize how badly the fibers were affected. The recovery studies at RT are shown as intensity and RIA spectra for the 50 kGy radiation exposure in Figure 4.8. The recovery rates at elevated temperatures are expected to be higher than the recovery rate at RT because more color centers start losing their stability as the temperature increases.

Figure 4.8.a-d. reveal that all the fiber samples were significantly affected and their light transmission abilities deteriorated. However, the fibers were started to recover from this radiation effect as time passes. It can be seen that the light intensities almost recovered completely for the wavelengths greater than 800 nm after three weeks. Therefore, it can be inferred that the color centers whose OA bands above 800 nm recovered at RT. In addition, the fibers could barely recover for the UV and visible ranges, which means that temperature healing itself is not enough for the mitigation of color centers in these ranges. Furthermore, the recovery rate seems to slow down as time passes which can be interpreted that there is a limited amount of color centers that can be mitigated at RT and their number decreases with time. Figure 4.8.e-h. shows the RIA spectra of the samples

was constructed using formula (2.23) for the intensity spectrums in Figure 4.8.a-d. RIA decreases with time and almost vanishes for greater wavelengths after three weeks. The technical difficulties caused the missing second week measurements for Fiber 1.

Fiber 1 experienced a significant light intensity loss when it was exposed to 50 kGy gamma radiation. It started to recover and re-gain its light transmission abilities after 1 week and recovered further at the end of week 3. However, this recovery was mostly in the NIR region and a little bit in the visible region, as shown in Figure 4.8.a. The rate of recovery was much lower in the UV region. This can be interpreted as the color centers whose OA bands reside in the UV region can not be thermally bleached efficiently at RT. The RIA recovery spectra in Figure 4.8.e. reveals this thermal bleaching behavior.

Fiber 2 and 3 exhibited quite similar recovery behaviors. Figure 4.8.b.&c. show that they went through immense radiation damage and partially recovered, which took place gradually over three weeks. Although this recovery was almost complete in the NIR region, it was partially effective in UV and visible regions where the RIA was still around 5 dB/m after week 3 as shown in Figure 4.8.f.&g.

The recovery of Fiber 4 occurred in NIR and partially in UV and visible regions as well. Figure 4.8.h. shows that the RIA values around 500 nm were 5 dB/m, comparable to other fibers. Therefore, it can be claimed that the home-made fibers behaved similarly to the commercial fiber in terms of radiation recovery, although some of them were affected more severely.

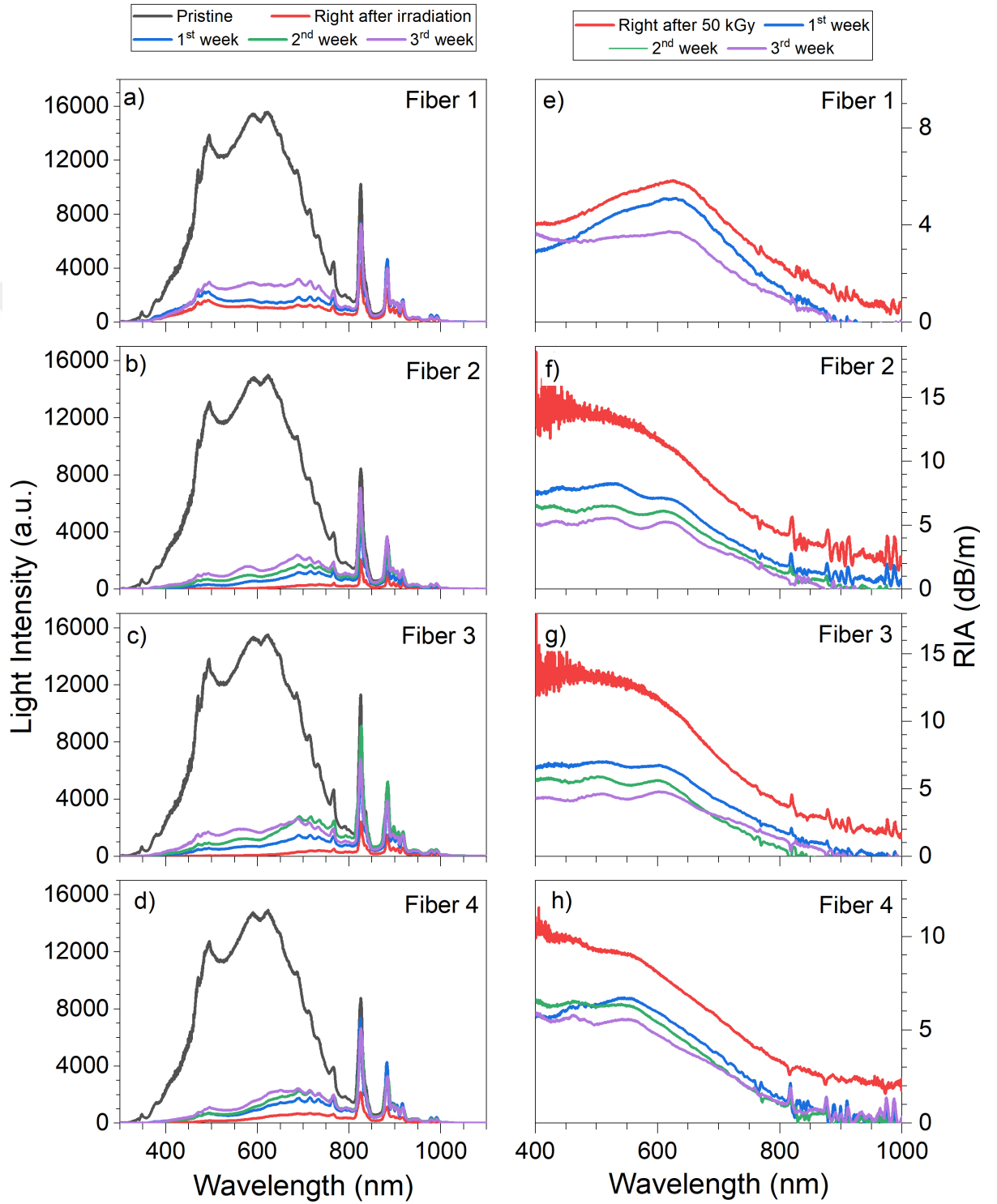


Figure 4.8: a-d) Light intensity recovery of Fiber 1, 2, 3 and 4. e-h) RIA recovery of Fiber 1, 2, 3 and 4 right after after gamma irradiation and three subsequent weeks

4.3 NA Measurements

NAs of the pristine and 50 kGy-irradiated samples of all four fibers were measured and the changes were obtained by software whose working principle depends on the formula 2.6. The changes that occurred in fiber NAs are given in Table 4.4.

Table 4.4: NA changes in fibers after radiation exposure

	Fiber 1	Fiber 2	Fiber 3	Fiber 4
Change	+11.1%	+34.2%	+8.4%	+1.5%

After radiation exposure, the increases in fiber NAs show that the RI differences between the fiber core and cladding increased as the formula (2.6) suggests. Considering that the $\sim 95\%$ of the core and 100% of the cladding is made of silica, the newly formed radiation induced Al and P related color centers in the fiber core must have acted in a way that the RI difference between the core and the cladding has increased. This radiation induced NA increase is in accordance with the works in literature in which radiation induced refractive index increase is observed in Yb-doped fibers as well as in fibers having various compositions. [97–101]

Although Fiber 2 and 3 were the most affected samples and showed a similar RIA behavior, their NA change values are quite different. Therefore, a direct correlation between the severity of the radiation damage and NA change can not be made.

Chapter 5

Conclusion

The effects of ionizing radiation like gamma rays or X-rays on optical fibers were studied in this thesis. Four Yb-doped optical fibers with different compositions were used to reveal gamma radiation's effect on their light transmission properties. RIA, color center growth and recovery measurements showed that the chemical composition of the fiber plays a crucial role in radiation resistance. The RE ions, Yb in this case, which are doped in the fiber core, are the most critical element that leads to RIA in optical fibers, which increases with the Yb mol%. The addition of other elements like Al and P also has a significant effect. They cause the formation of additional color centers and an increase in RIA; however, their relative ratio to each other as well as to Yb might act as an agent that decreases the radiation damage; hence, RIA and is crucial for radiation resistance. The fabrication processes have become very important since they are the steps that give the fibers their final properties. The Al and P should be doped into fiber in precise amounts so that the Yb can be soluble in silica and there can be enough distance between Yb ions to prevent their clustering and interactions between them. The P/Al and Al/Yb ratios should be arranged in a manner that the fibers have desired guiding properties as well as a composition that can resist radiation damage. For fiber dosimetry studies, an operation wavelength should be chosen such that the color centers within that particular range can be fully characterized and be recoverable for subsequent uses. The less the number of

color centers, the more straightforward fiber dosimeter operation; therefore, UV and visible wavelengths do not appear to be a good option since there are a lot of various color centers that are not fully recoverable. However, phosphorus-related P_1 defect whose OA band peaks at around 1570 nm lays on IR range all by itself and seems to be a good fit for this purpose.

In order to obtain more precise and accurate results about the RIA in fibers, some additional characterization techniques should be performed. Some experiments that utilized Raman spectroscopy and Fourier-transform infrared spectroscopy (FTIR) were conducted; however, precise and reproducible data explain the effects of gamma radiation on Yb-doped optical fibers could not be obtained. More careful and elaborate experiments with these devices can enhance the results. Moreover, Electron Spin Resonance (ESR) spectroscopy, solid-state Nuclear Magnetic Resonance (NMR) spectroscopy, and mass spectroscopy (MS) techniques give quite detailed and exact results. Therefore, the fiber itself and the defects created by the radiation can be characterized without any uncertainties. These measurements could not be performed due to the lack of equipment. Another suggestion can be the use of fibers with just one changing parameter in their composition so that the effect of each element to RIA can be understood better and more straightforward.

For future work, an optical setup used for photobleaching studies should be utilized to understand which types of defects can be recovered and to what extent by treating the radiation-damaged fibers with high-energy photons. The suggested techniques and the photobleaching studies have great importance and they are crucial for the fabrication of radiation-resistant fibers and fiber dosimetries.

Bibliography

- [1] J. Hecht, *City of Light: The story of fiber optics*. Oxford University Press, 2004.
- [2] A. Hideur, T. Chartier, C. Özkul, and F. Sanchez, “All-fiber tunable ytterbium-doped double-clad fiber ring laser,” *Optics Letters*, vol. 26, no. 14, pp. 1054–1056, 2001.
- [3] Y. e. Jeong, J. Sahu, D. Payne, and J. Nilsson, “Ytterbium-doped large-core fiber laser with 1.36 kW continuous-wave output power,” *Optics express*, vol. 12, no. 25, pp. 6088–6092, 2004.
- [4] V. Dominic, S. MacCormack, R. Waarts, S. Sanders, S. Bicknese, R. Dohle, E. Wolak, P. Yeh, and E. Zucker, “110 W fibre laser,” *Electronics Letters*, vol. 35, no. 14, pp. 1158–1160, 1999.
- [5] A. Tünnermann, T. Schreiber, F. Röser, A. Liem, S. Höfer, H. Zellmer, S. Nolte, and J. Limpert, “The renaissance and bright future of fibre lasers,” *Journal of Physics B: Atomic, Molecular and Optical Physics*, vol. 38, no. 9, p. S681, 2005.
- [6] D. J. Richardson, J. Nilsson, and W. A. Clarkson, “High power fiber lasers: Current status and future perspectives,” *JOSA B*, vol. 27, no. 11, pp. B63–B92, 2010.
- [7] B. He, J. Zhou, Q. Lou, Y. Xue, Z. Li, W. Wang, J. Dong, Y. Wei, and W. Chen, “1.75-kilowatt continuous-wave output fiber laser using home-made ytterbium-doped large-core fiber,” *Microwave and Optical Technology Letters*, vol. 52, no. 7, pp. 1668–1671, 2010.

- [8] L. Zhang, G. Li, W. Li, Q. Gao, Z. Li, W. Zhao, B. Zhao, and C. Hou, “kW-level low photodarkening Yb/Ce codoped aluminosilicate fiber fabricated by the chelate gas phase deposition technique,” *Optical Materials Express*, vol. 6, no. 11, pp. 3558–3564, 2016.
- [9] S. Girard, J. Keurinck, A. Boukenter, J.-P. Meunier, Y. Ouerdane, B. Azais, P. Charre, and M. Vié, “Gamma-rays and pulsed x-ray radiation responses of nitrogen-, germanium-doped and pure silica core optical fibers,” *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, vol. 215, no. 1-2, pp. 187–195, 2004.
- [10] S. Agnello, D. Di Francesca, A. Alessi, G. Iovino, M. Cannas, S. Girard, A. Boukenter, and Y. Ouerdane, “Interstitial O₂ distribution in amorphous SiO₂ nanoparticles determined by raman and photoluminescence spectroscopy,” *Journal of Applied Physics*, vol. 114, no. 10, p. 104305, 2013.
- [11] B. Fox, Z. Schneider, K. Simmons-Potter, W. Thomes Jr, D. Meister, R. Bambha, and D. Kliner, “Gamma radiation effects in Yb-doped optical fiber,” in *Fiber Lasers IV: Technology, Systems, and Applications*, vol. 6453, p. 645328, International Society for Optics and Photonics, 2007.
- [12] G. L. Vecchi, D. Di Francesca, C. Sabatier, S. Girard, A. Alessi, A. Guttilla, T. Robin, Y. Kadi, and M. Brugger, “Infrared radiation induced attenuation of radiation sensitive optical fibers: Influence of temperature and modal propagation,” *Optical Fiber Technology*, vol. 55, p. 102166, 2020.
- [13] C. Shao, J. Ren, F. Wang, N. Ollier, F. Xie, X. Zhang, L. Zhang, C. Yu, and L. Hu, “Origin of radiation-induced darkening in Yb³⁺/Al³⁺/P³⁺-doped silica glasses: Effect of the P/Al ratio,” *The Journal of Physical Chemistry B*, vol. 122, no. 10, pp. 2809–2820, 2018.
- [14] R.-x. Xing, Y.-b. Sheng, Z.-j. Liu, H.-q. Li, Z.-w. Jiang, J.-g. Peng, L.-y. Yang, J.-y. Li, and N.-l. Dai, “Investigation on radiation resistance of Er/Ce co-doped silicate glasses under 5 kGy gamma-ray irradiation,” *Optical Materials Express*, vol. 2, no. 10, pp. 1329–1335, 2012.

- [15] S. Girard, J. Kuhnhenh, A. Gusarov, B. Brichard, M. Van Uffelen, Y. Ouerdane, A. Boukenter, and C. Marcandella, “Radiation effects on silica-based optical fibers: Recent advances and future challenges,” *IEEE Transactions on Nuclear Science*, vol. 60, no. 3, pp. 2015–2036, 2013.
- [16] D. Sporea, A. Sporea, S. O’Keeffe, D. McCarthy, and E. Lewis, “Optical fibers and optical fiber sensors used in radiation monitoring,” in *Selected Topics on Optical Fiber Technology* (M. Yasin, S. W. Harun, and H. Arof, eds.), ch. 23, Rijeka: IntechOpen, 2012.
- [17] C. Hoehr, M. Hanna, S. Zeisler, C. Penner, M. Stokely, and M. Dehnel, “Ce-and B-doped silica fibers for monitoring low-energy proton beams on a medical cyclotron,” *Applied Sciences*, vol. 10, no. 13, p. 4488, 2020.
- [18] M. Bakaic, M. Hanna, C. Hnatovsky, D. Grobnc, S. Mihailov, S. S. Zeisler, and C. Hoehr, “Fiber-optic bragg gratings for temperature and pressure measurements in isotope production targets for nuclear medicine,” *Applied Sciences*, vol. 10, no. 13, p. 4610, 2020.
- [19] C. Hoehr, A. Morana, O. Duhamel, B. Capoen, M. Trinczek, P. Paillet, C. Duzenli, M. Bouazaoui, G. Bouwmans, A. Cassez, *et al.*, “Novel Gd³⁺-doped silica-based optical fiber material for dosimetry in proton therapy,” *Scientific reports*, vol. 9, no. 1, pp. 1–8, 2019.
- [20] C. Penner, P. Woulfe, B. Stoeber, C. Duzenli, S. O’Keeffe, and C. Hoehr, “Novel optical fibre-based sensors for neutron and proton beams,” in *2019 IEEE SENSORS*, pp. 1–4, IEEE, 2019.
- [21] D. Lyu, J. Peng, Q. Huang, W. Zheng, L. Xiong, and M. Yang, “Radiation-resistant optical fiber Fabry-Perot interferometer used for high-temperature sensing,” *IEEE Sensors Journal*, vol. 21, no. 1, pp. 57–61, 2020.
- [22] S. Girard, A. Morana, C. Hoehr, M. Trinczek, J. Vidalot, P. Paillet, C. Bélanger-Champagne, J. Mekki, N. Balcon, G. Li Vecchi, *et al.*, “Atmospheric neutron monitoring through optical fiber-based sensing,” *Sensors*, vol. 20, no. 16, p. 4510, 2020.

- [23] Y. Midilli and B. Ortaç, “Demonstration of an all-fiber ultra-low numerical aperture ytterbium-doped large mode area fiber in a master oscillator power amplifier configuration above 1 kW power level,” *Journal of Light-wave Technology*, vol. 38, no. 7, pp. 1915–1920, 2020.
- [24] C. Jauregui, J. Limpert, and A. Tünnermann, “High-power fibre lasers,” *Nature photonics*, vol. 7, no. 11, pp. 861–867, 2013.
- [25] M.-J. Li, X. Chen, J. Wang, S. Gray, A. Liu, J. A. Demeritt, A. B. Ruffin, A. M. Crowley, D. T. Walton, and L. A. Zenteno, “Al/Ge co-doped large mode area fiber with high SBS threshold,” *Optics Express*, vol. 15, no. 13, pp. 8290–8299, 2007.
- [26] D. Nodop, C. Jauregui, F. Jansen, J. Limpert, and A. Tünnermann, “Suppression of stimulated raman scattering employing long period gratings in double-clad fiber amplifiers,” *Optics letters*, vol. 35, no. 17, pp. 2982–2984, 2010.
- [27] J. Kim, P. Dupriez, C. Codemard, J. Nilsson, and J. Sahu, “Suppression of stimulated Raman scattering in a high power Yb-doped fiber amplifier using a W-type core with fundamental mode cut-off,” *Optics express*, vol. 14, no. 12, pp. 5103–5113, 2006.
- [28] R. Stolen and C. Lin, “Self-phase-modulation in silica optical fibers,” *Physical Review A*, vol. 17, no. 4, p. 1448, 1978.
- [29] R. L. Farrow, D. A. Kliner, G. R. Hadley, and A. V. Smith, “Peak-power limits on fiber amplifiers imposed by self-focusing,” *Optics letters*, vol. 31, no. 23, pp. 3423–3425, 2006.
- [30] D. J. Richardson, J. Nilsson, and W. A. Clarkson, “High power fiber lasers: current status and future perspectives,” *JOSA B*, vol. 27, no. 11, pp. B63–B92, 2010.
- [31] T. Eidam, C. Wirth, C. Jauregui, F. Stutzki, F. Jansen, H.-J. Otto, O. Schmidt, T. Schreiber, J. Limpert, and A. Tünnermann, “Experimental observations of the threshold-like onset of mode instabilities in high power fiber amplifiers,” *Optics express*, vol. 19, no. 14, pp. 13218–13224, 2011.

- [32] C. Jauregui, T. Eidam, J. Limpert, and A. Tünnermann, “Impact of modal interference on the beam quality of high-power fiber amplifiers,” *Optics express*, vol. 19, no. 4, pp. 3258–3271, 2011.
- [33] A. V. Smith and J. J. Smith, “Mode instability in high power fiber amplifiers,” *Optics express*, vol. 19, no. 11, pp. 10180–10192, 2011.
- [34] T. Hadjifotiou, “Optical fibers,” in *The Cable and Telecommunications Professionals’ Reference (Third Edition)* (G. Hill, ed.), pp. 111 – 144, Boston: Focal Press, third edition ed., 2008.
- [35] E. Hecht, *Optics*. Pearson, 2016.
- [36] G. P. Agrawal, *Fiber-optic communication systems*. John Wiley & Sons, 2012.
- [37] S. Sudo and H. Itoh, “Efficient non-linear optical fibres and their applications,” *Optical and quantum electronics*, vol. 22, no. 3, pp. 187–212, 1990.
- [38] C. Yeh, “Introduction to the optical fiber,” in *Handbook of Fiber Optics* (C. Yeh, ed.), p. 11, San Diego: Academic Press, 1990.
- [39] A. W. Snyder and J. Love, *Optical waveguide theory*. Springer Science & Business Media, 2012.
- [40] P. A. Tick and P. L. Bocko, “Optical fiber materials,” in *Optical Materials: A Series of Advances* (S. Musikant, ed.), pp. 147–260, New York: Marcel Dekker, 1990.
- [41] E. Kendir, *Kuartz optik fiberlerin ve Ce katkılı sintilasyon fiberlerin radyasyon altında optik ve yapısal özelliklerinin incelenmesi*. PhD thesis, Akdeniz University, Antalya, 2019.
- [42] P. D. Dragic, M. Cavillon, and J. Ballato, “Materials for optical fiber lasers: A review,” *Applied Physics Reviews*, vol. 5, no. 4, p. 041301, 2018.
- [43] P. Jelger, M. Engholm, L. Norin, and F. Laurell, “Degradation-resistant lasing at 980 nm in a Yb/Ce/Al-doped silica fiber,” *JOSA B*, vol. 27, no. 2, pp. 338–342, 2010.

- [44] S. Unger, F. Lindner, C. Aichele, M. Leich, A. Schwuchow, J. Kobelke, J. Dellith, K. Schuster, and H. Bartelt, “A highly efficient Yb-doped silica laser fiber prepared by gas phase doping technology,” *Laser Physics*, vol. 24, no. 3, p. 035103, 2014.
- [45] J. Koerner, C. Vorholt, H. Liebetrau, M. Kahle, D. Kloepfel, R. Seifert, J. Hein, and M. C. Kaluza, “Measurement of temperature-dependent absorption and emission spectra of Yb:YAG, Yb:LuAG, and Yb:CaF₂ between 20 °C and 200 °C and predictions on their influence on laser performance,” *JOSA B*, vol. 29, no. 9, pp. 2493–2502, 2012.
- [46] D. F. Swinehart, “The Beer-Lambert law,” *Journal of chemical education*, vol. 39, no. 7, p. 333, 1962.
- [47] J. MacChesney, P. O’connor, and H. Presby, “A new technique for the preparation of low-loss and graded-index optical fibers,” *Proceedings of the IEEE*, vol. 62, no. 9, pp. 1280–1281, 1974.
- [48] S. M. Yassin, N. Omar, and H. Abdul-Rashid, “Optimized fabrication of thulium doped silica optical fiber using MCVD,” in *Handbook of Optical Fibers*, pp. 1–35, Springer Singapore, 2018.
- [49] S. R. Nagel, J. B. MacChesney, and K. L. Walker, “An overview of the modified chemical vapor deposition (MCVD) process and performance,” *IEEE Transactions on Microwave Theory and Techniques*, vol. 30, no. 4, pp. 305–322, 1982.
- [50] S. Nagel, J. MacChesney, and K. Walker, “Modified chemical vapor deposition,” in *Optical Fiber Communications* (T. Li, ed.), pp. 1 – 64, Academic Press, 1985.
- [51] D. Lipatov, A. Guryanov, M. Yashkov, M. Bubnov, and M. Likhachev, “Fabrication of Yb₂O₃–Al₂O₃–P₂O₅–SiO₂ optical fibers with a perfect step-index profile by the MCVD process,” *Inorganic Materials*, vol. 54, no. 3, pp. 276–282, 2018.

- [52] T. Edahiro, M. Horiguchi, K. Chida, and Y. Ohmori, "Spectral loss characteristics of GeO_2 - P_2O_5 -doped silica graded-index fibres in long-wavelength band," *Electronics Letters*, vol. 15, no. 10, pp. 274–275, 1979.
- [53] D. L. Wood, T. Y. Kometani, J. P. Luongo, and M. A. Saifi, "Incorporation of OH in Glass in the MCVD Process," *Journal of the American Ceramic Society*, vol. 62, no. 11-12, pp. 638–639, 1979.
- [54] D. L. Wood and J. S. Shirk, "Partition of hydrogen in the modified chemical vapor deposition process," *Journal of the American Ceramic Society*, vol. 64, no. 6, pp. 325–327, 1981.
- [55] K. L. Walker, J. B. Macchesney, and J. R. Simpson, "Reduction of hydroxyl contamination in optical fiber preforms," in *Integrated Optics and Optical Fiber Communication*, p. WA4, Optical Society of America, 1981.
- [56] D. J. DiGiovanni, R. Shubochkin, T. Morse, and B. Lenardic, "Rare earth-doped fibers," in *Specialty Optical Fibers Handbook* (A. Méndez and T. Morse, eds.), pp. 195 – 242, Burlington: Academic Press, 2007.
- [57] H. Henschel, O. Kohn, H. Schmidt, J. Kirchhof, and S. Unger, "Radiation-induced loss of rare earth doped silica fibres," in *RADECS 97. Fourth European Conference on Radiation and its Effects on Components and Systems (Cat. No. 97TH8294)*, pp. 439–444, IEEE, 1997.
- [58] S. Girard, Y. Ouerdane, G. Origlio, C. Marcandella, A. Boukenter, N. Richard, J. Baggio, P. Paillet, M. Cannas, J. Bisutti, *et al.*, "Radiation effects on silica-based preforms and optical fibers—I: Experimental study with canonical samples," *IEEE Transactions on Nuclear Science*, vol. 55, no. 6, pp. 3473–3482, 2008.
- [59] S. Girard, Y. Ouerdane, C. Marcandella, A. Boukenter, S. Quenard, and N. Authier, "Feasibility of radiation dosimetry with phosphorus-doped optical fibers in the ultraviolet and visible domain," *Journal of non-crystalline solids*, vol. 357, no. 8-9, pp. 1871–1874, 2011.

- [60] S. Girard, A. Morana, A. Ladaci, T. Robin, L. Mescia, J.-J. Bonnefois, M. Boutillier, J. Mekki, A. Paveau, B. Cadier, *et al.*, “Recent advances in radiation-hardened fiber-based technologies for space applications,” *Journal of Optics*, vol. 20, no. 9, p. 093001, 2018.
- [61] S. Girard, A. Alessi, N. Richard, L. Martin-Samos, V. De Michele, L. Giacomazzi, S. Agnello, D. Di Francesca, A. Morana, B. Winkler, *et al.*, “Overview of radiation induced point defects in silica-based optical fibers,” *Reviews in Physics*, vol. 4, p. 100032, 2019.
- [62] L. Skuja, “Optically active oxygen-deficiency-related centers in amorphous silicon dioxide,” *Journal of NON-crystalline Solids*, vol. 239, no. 1-3, pp. 16–48, 1998.
- [63] D. L. Griscom, E. Friebele, K. Long, and J. Fleming, “Fundamental defect centers in glass: Electron spin resonance and optical absorption studies of irradiated phosphorus-doped silica glass and optical fibers,” *Journal of Applied Physics*, vol. 54, no. 7, pp. 3743–3762, 1983.
- [64] D. L. Griscom, “The natures of point defects in amorphous silicon dioxide,” in *Defects in SiO₂ and related dielectrics: Science and technology*, pp. 117–159, Springer, 2000.
- [65] N. Akchurin, E. Kendir, Ş. Yaltkaya, J. Damgov, F. De Guio, and S. Kunori, “Radiation-hardness studies with cerium-doped fused-silica fibers,” *Journal of Instrumentation*, vol. 14, no. 03, p. P03020, 2019.
- [66] E. Regnier, I. Flammer, S. Girard, F. Gooijer, F. Achten, and G. Kuyt, “Low-dose radiation-induced attenuation at infrared wavelengths for P-doped, Ge-doped and pure silica-core optical fibres,” *IEEE transactions on nuclear science*, vol. 54, no. 4, pp. 1115–1119, 2007.
- [67] D. Di Francesca, S. Girard, S. Agnello, A. Alessi, C. Marcandella, P. Paillet, Y. Ouerdane, Y. Kadi, M. Brugger, and A. Boukenter, “Combined temperature radiation effects and influence of drawing conditions on phosphorous-doped optical fibers,” *physica status solidi (a)*, vol. 216, no. 3, p. 1800553, 2019.

- [68] S. Girard, C. Marcandella, A. Morana, J. Perisse, D. Di Francesca, P. Paillet, J.-R. Macé, A. Boukenter, M. León, M. Gaillardin, *et al.*, “Combined high dose and temperature radiation effects on multimode silica-based optical fibers,” *IEEE transactions on nuclear science*, vol. 60, no. 6, pp. 4305–4313, 2013.
- [69] D. Di Francesca, S. Girard, S. Agnello, A. Alessi, C. Marcandella, P. Paillet, N. Richard, A. Boukenter, Y. Ouerdane, and F. Gelardi, “Radiation response of Ce-codoped germanosilicate and phosphosilicate optical fibers,” *IEEE Transactions on Nuclear Science*, vol. 63, no. 4, pp. 2058–2064, 2016.
- [70] E. J. Friebele and M. E. Gingerich, “Photobleaching effects in optical fiber waveguides,” *Applied optics*, vol. 20, no. 19, pp. 3448–3452, 1981.
- [71] C. Shao, Y. Jiao, F. Lou, M. Wang, L. Zhang, S. Feng, S. Wang, D. Chen, C. Yu, and L. Hu, “Enhanced radiation resistance of ytterbium-doped silica fiber by pretreating on a fiber preform,” *Optical Materials Express*, vol. 10, no. 2, pp. 408–420, 2020.
- [72] Y. Xing, Y. Liu, R. Cao, L. Liao, Y. Chu, Y. Wang, J. Peng, H. Li, L. Yang, N. Dai, *et al.*, “Elimination of radiation damage in Tm-doped silica fibers based on the radical bleaching of deuterium loading,” *OSA Continuum*, vol. 1, no. 3, pp. 987–995, 2018.
- [73] D. L. Griscom, “Fractal kinetics of radiation-induced point-defect formation and decay in amorphous insulators: Application to color centers in silica-based optical fibers,” *Physical Review B*, vol. 64, no. 17, p. 174201, 2001.
- [74] L. Skuja, “Optical properties of defects in silica,” in *Defects in SiO₂ and related dielectrics: Science and technology*, pp. 73–116, Springer, 2000.
- [75] D. L. Griscom, “Optical properties and structure of defects in silica glass,” *Journal of the Ceramic Society of Japan*, vol. 99, no. 1154, pp. 923–942, 1991.
- [76] L. Skuja, “Isoelectronic series of twofold coordinated Si, Ge, and Sn atoms in glassy SiO₂:A luminescence study,” *Journal of non-crystalline solids*, vol. 149, no. 1-2, pp. 77–95, 1992.

- [77] L. Skuja, “The origin of the intrinsic 1.9 eV luminescence band in glassy SiO₂,” *Journal of Non-Crystalline Solids*, vol. 179, pp. 51–69, 1994.
- [78] L. Skuja, K. Tanimura, and N. Itoh, “Correlation between the radiation-induced intrinsic 4.8 eV optical absorption and 1.9 eV photoluminescence bands in glassy SiO₂,” *Journal of applied physics*, vol. 80, no. 6, pp. 3518–3525, 1996.
- [79] C. Sousa, C. de Graaf, and G. Pacchioni, “Optical properties of peroxy radicals in silica: Multiconfigurational perturbation theory calculations,” *The Journal of Chemical Physics*, vol. 114, no. 14, pp. 6259–6264, 2001.
- [80] D. Donadio, M. Bernasconi, and M. Boero, “Ab initio simulations of photoinduced interconversions of oxygen deficient centers in amorphous silica,” *Physical review letters*, vol. 87, no. 19, p. 195504, 2001.
- [81] B. Winkler, L. Martin-Samos, N. Richard, L. Giacomazzi, A. Alessi, S. Girard, A. Boukenter, Y. Ouerdane, and M. Valant, “Correlations between structural and optical properties of peroxy bridges from first principles,” *The Journal of Physical Chemistry C*, vol. 121, no. 7, pp. 4002–4010, 2017.
- [82] L. Skuja, K. Kajihara, T. Kinoshita, M. Hirano, and H. Hosono, “The behavior of interstitial oxygen atoms induced by F₂ laser irradiation of oxygen-rich glassy SiO₂,” *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, vol. 191, no. 1-4, pp. 127–130, 2002.
- [83] N. Kuzuu and H. Horikoshi, “X-ray induced absorption in fused silica containing various amounts of OH,” *Journal of applied physics*, vol. 97, no. 9, p. 093508, 2005.
- [84] D. L. Griscom, “Self-trapped holes in amorphous silicon dioxide,” *Physical Review B*, vol. 40, no. 6, p. 4224, 1989.
- [85] D. L. Griscom, “Electron spin resonance characterization of self-trapped holes in amorphous silicon dioxide,” *Journal of non-crystalline solids*, vol. 149, no. 1-2, pp. 137–160, 1992.

- [86] D. L. Griscom, “Self-trapped holes in pure-silica glass: A history of their discovery and characterization and an example of their critical significance to industry,” *Journal of non-crystalline solids*, vol. 352, no. 23-25, pp. 2601–2617, 2006.
- [87] J. Stone and G. E. Walrafen, “Overtone vibrations of OH groups in fused silica optical fibers,” *The Journal of Chemical Physics*, vol. 76, no. 4, pp. 1712–1722, 1982.
- [88] D. Ehrt, P. Ebeling, and U. Natura, “UV Transmission and radiation-induced defects in phosphate and fluoride-phosphate glasses,” *Journal of Non-Crystalline Solids*, vol. 263, pp. 240–250, 2000.
- [89] T. Deschamps, N. Ollier, H. Vezin, and C. Gonnet, “Clusters dissolution of Yb^{3+} in codoped $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-P}_2\text{O}_5$ glass fiber and its relevance to photodarkening,” *The Journal of Chemical Physics*, vol. 136, no. 1, p. 014503, 2012.
- [90] D. Di Francesca, S. Girard, S. Agnello, A. Alessi, C. Marcandella, P. Paillet, Y. Ouerdane, Y. Kadi, M. Brugger, and A. Boukenter, “Combined temperature radiation effects and influence of drawing conditions on phosphorous-doped optical fibers,” *physica status solidi (a)*, vol. 216, no. 3, p. 1800553, 2019.
- [91] H. Hideo and K. Hiroshi, “Radiation-induced coloring and paramagnetic centers in synthetic $\text{SiO}_2\text{: Al}$ glasses,” *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, vol. 91, no. 1-4, pp. 395–399, 1994.
- [92] M. Engholm, L. Norin, and D. Åberg, “Strong UV absorption and visible luminescence in ytterbium-doped aluminosilicate glass under UV excitation,” *Optics letters*, vol. 32, no. 22, pp. 3352–3354, 2007.
- [93] M. Engholm and L. Norin, “Preventing photodarkening in ytterbium-doped high power fiber lasers; correlation to the UV-transparency of the core glass,” *Optics Express*, vol. 16, no. 2, pp. 1260–1268, 2008.

- [94] S. Rydberg and M. Engholm, “Experimental evidence for the formation of divalent ytterbium in the photodarkening process of Yb-doped fiber lasers,” *Optics express*, vol. 21, no. 6, pp. 6681–6688, 2013.
- [95] S. Wang, F. Lou, C. Yu, Q. Zhou, M. Wang, S. Feng, D. Chen, L. Hu, W. Chen, M. Guzik, and G. Boulon, “Influence of Al^{3+} and P^{5+} ion contents on the valence state of Yb^{3+} ions and the dispersion effect of Al^{3+} and P^{5+} ions on Yb^{3+} ions in silica glass,” *J. Mater. Chem. C*, vol. 2, pp. 4406–4414, 2014.
- [96] F. Wang, C. Shao, C. Yu, S. Wang, L. Zhang, G. Gao, and L. Hu, “Effect of AlPO_4 join concentration on optical properties and radiation hardening performance of Yb-doped Al_2O_3 - P_2O_5 - SiO_2 glass,” *Journal of Applied Physics*, vol. 125, no. 17, p. 173104, 2019.
- [97] M. Tao, H. Chen, G. Feng, K. Luan, F. Wang, K. Huang, and X. Ye, “Thermal modeling of high-power Yb-doped fiber lasers with irradiated active fibers,” *Optics Express*, vol. 28, no. 7, pp. 10104–10123, 2020.
- [98] A. Vanderka, L. Bednarek, L. Hajek, J. Latal, R. Poboril, P. Zavodny, and V. Vasinek, “Measurement of the optical fiber numeric aperture exposed to thermal and radiation aging,” in *20th Slovak-Czech-Polish Optical Conference on Wave and Quantum Aspects of Contemporary Optics*, vol. 10142, p. 1014224, International Society for Optics and Photonics, 2016.
- [99] M. Medhat, S. El-Zaiat, S. M. Abdou, A. Radi, and M. Omar, “Interferometric determination of gamma radiation effects on optical parameters of a GRIN optical fibre,” *Journal of Optics A: Pure and Applied Optics*, vol. 4, no. 4, p. 485, 2002.
- [100] A. F. Fernandez, B. Brichard, and F. Berghmans, “In situ measurement of refractive index changes induced by gamma radiation in germanosilicate fibers,” *IEEE Photonics Technology Letters*, vol. 15, no. 10, pp. 1428–1430, 2003.

- [101] S. Kher, S. Chaubey, S. Oak, and A. Gusarov, “Measurement of γ -radiation induced refractive index changes in B/Ge doped fiber using LPGs,” *IEEE Photonics Technology Letters*, vol. 25, no. 21, pp. 2070–2073, 2013.

